Metallographic study of phase changes in heat treated ductile iron

Mahendra Vadilal Desai

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METALLOGRAPHIC STUDY OF PHASE CHANGES IN HEAT TREATED DUCTILE IRON

BY
MAHENDRA VADILAL DESAI, 1943-

A
THESIS
submitted to the faculty of
UNIVERSITY OF MISSOURI - ROLLA
in partial fulfillment of the requirements for the
Degree of
MASTER OF SCIENCE IN METALLURGICAL ENGINEERING
Rolla, Missouri
1970

Approved by

[Signature]

[Signature]
Continuous cast ductile iron samples were hardened. It was found that carbide dissolved in the austenite faster than graphite and that this material requires higher austenitizing temperatures and longer austenitizing time than steels. Tempered microstructures were affected by the prior austenitizing treatment and it appeared that the amount of carbon dissolved in the austenite determined the tempered microstructure.

Several attempts to determine the austenite grain size indicated that it is not as easy to determine in ductile iron as in steels. Two techniques, heat etching and an isothermal transformation method, were found to reveal prior austenite grain size.

It was concluded that carbon precipitated directly as graphite from martensite in ductile iron rather than as combined carbon the way it does in steels. The reason for direct precipitation of graphite from martensite was not known.

Repeated quenching and subsequent tempering might lead to the replacement of the primary graphite dispersion by a secondary graphite dispersion. This could alter properties but it did not appear to be a practical process because of the large number of quenching and tempering cycles required.

A relationship between regular Rockwell C hardness and the matrix hardness was established which was in good
agreement with that in the literature. An approximate partial isothermal transformation diagram is reported.
ACKNOWLEDGEMENT

The author wishes to express his gratitude to Dr. Fred Kisslinger for his guidance, advice, and assistance in the execution of this work and preparation of this thesis.

The author is indebted to Wells Manufacturing Co., Skokie, Illinois, for their generosity in supplying continuous cast ductile iron for this investigation.

The assistance and cooperation of the other faculty members and graduate students is gratefully acknowledged.
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>List of Figures</td>
<td>vii</td>
</tr>
<tr>
<td>List of Tables</td>
<td>xii</td>
</tr>
<tr>
<td>I. Introduction</td>
<td>1</td>
</tr>
<tr>
<td>II. Literature Review</td>
<td>2</td>
</tr>
<tr>
<td>III. Experimental Procedure</td>
<td>6</td>
</tr>
<tr>
<td>A. Material Studied</td>
<td>8</td>
</tr>
<tr>
<td>B. Heat Treating</td>
<td>9</td>
</tr>
<tr>
<td>C. Specimen Preparation</td>
<td>13</td>
</tr>
<tr>
<td>D. Metallographic Studies</td>
<td>13</td>
</tr>
<tr>
<td>E. Hardness Tests</td>
<td>13</td>
</tr>
<tr>
<td>IV. Results and Discussions</td>
<td>15</td>
</tr>
<tr>
<td>A. Hardening of Ductile Iron</td>
<td>15</td>
</tr>
<tr>
<td>B. Austenite Grain Size</td>
<td>34</td>
</tr>
<tr>
<td>1. Gradient Quench Method</td>
<td>41</td>
</tr>
<tr>
<td>2. Vilella's Method</td>
<td>43</td>
</tr>
<tr>
<td>3. Oxidation Method</td>
<td>45</td>
</tr>
<tr>
<td>4. Heat Etching</td>
<td>47</td>
</tr>
<tr>
<td>5. Isothermal Transformation</td>
<td>55</td>
</tr>
<tr>
<td>6. Variation of Tempered Microstructures</td>
<td>56</td>
</tr>
<tr>
<td>C. Precipitation of Carbon from Martensite</td>
<td>62</td>
</tr>
<tr>
<td>1. Tempering of Quenched Ductile Iron</td>
<td>63</td>
</tr>
<tr>
<td>a. Samples Tempered at 1250°F</td>
<td>63</td>
</tr>
<tr>
<td>b. Samples Tempered at 1000°F</td>
<td>69</td>
</tr>
<tr>
<td>c. Samples Tempered at 700°F and 400°F.</td>
<td>72</td>
</tr>
<tr>
<td>d. Discussion</td>
<td>72</td>
</tr>
<tr>
<td>Section</td>
<td>Page</td>
</tr>
<tr>
<td>--------------------------------------------------</td>
<td>------</td>
</tr>
<tr>
<td>2. Tempering of Normalized Ductile Iron</td>
<td>75</td>
</tr>
<tr>
<td>3. Isothermal Transformation of Ductile Iron</td>
<td>78</td>
</tr>
<tr>
<td>a. Isothermal Transformation at 1300°F</td>
<td>80</td>
</tr>
<tr>
<td>b. Isothermal Transformation at 1200°F</td>
<td>84</td>
</tr>
<tr>
<td>c. Isothermal Transformation at 1100°F</td>
<td>89</td>
</tr>
<tr>
<td>d. Isothermal Transformation at 1000°F</td>
<td>94</td>
</tr>
<tr>
<td>e. Isothermal Transformation at 900°F</td>
<td>99</td>
</tr>
<tr>
<td>4. Discussion</td>
<td>104</td>
</tr>
<tr>
<td>D. Repeated Quenching and Tempering</td>
<td>117</td>
</tr>
<tr>
<td>E. Relationship Between the Hardness Readings</td>
<td>121</td>
</tr>
<tr>
<td>F. Isothermal Transformation Diagram</td>
<td>123</td>
</tr>
<tr>
<td>V. CONCLUSIONS</td>
<td>125</td>
</tr>
<tr>
<td>BIBLIOGRAPHY</td>
<td>127</td>
</tr>
<tr>
<td>VITA</td>
<td>129</td>
</tr>
</tbody>
</table>
LIST OF FIGURES

Fig. 1 As-received sample of ductile iron which shows spheroidal primary graphite in a ferritic matrix .......................... 11

Fig. 2 As-received sample of ductile iron which shows spheroidal and vermicular primary graphite in a ferritic matrix .................. 11

Fig. 3 As-quenched hardness as a function of austenitizing temperature .......................................................... 18

Fig. 4 The hardness of martensite as a function of carbon content ................................................................. 20

Fig. 5 As-quenched hardness as a function of austenitizing time for three different austenitizing temperatures .................. 23

Fig. 6 Sample austenitized at 1500°F for 2 hr., water quenched and tempered at 1250°F for 32 hr. .................. 25

Fig. 7 Sample austenitized at 1600°F for 2 hr., water quenched and tempered at 1250°F for 32 hr. .................. 26

Fig. 8 Sample austenitized at 1700°F for 2 hr., water quenched and tempered at 1250°F for 32 hr. .................. 27

Fig. 9 Sample austenitized at 1800°F for 2 hr., water quenched and tempered at 1250°F for 32 hr. .................. 27

Fig. 10 Sample austenitized at 1900°F for 2 hr., water quenched and tempered at 1250°F for 32 hr. .................. 28

Fig. 11 Sample austenitized at 1550°F for 4 hr., water quenched and tempered at 1250°F for 2 hr. .................. 30

Fig. 12 Sample austenitized at 1550°F for 10 hr., water quenched and tempered at 1250°F for 2 hr. .................. 30

Fig. 13 Sample austenitized at 1550°F for 44 hr., water quenched and tempered at 1250°F for 2 hr. .................. 31

Fig. 14 Sample austenitized at 1900°F for 15 min., water quenched and tempered at 1250°F for 2 hr. .................. 31

Fig. 15 Sample austenitized at 1900°F for 60 min., water quenched and tempered at 1250°F for 2 hr. .................. 32

Fig. 16 Sample austenitized at 1900°F for 2 hr., water quenched and tempered at 1250°F for 2 hr. .................. 32
Fig. 17 Grain size as a function of austenitizing temperature for a fine-grained type steel.

Fig. 18 Ductile iron sample of 0.2 inch diameter that had been gradient quenched from 1750°F.

Fig. 19 Ductile iron sample shown in Figure 18 in the unetched condition.

Fig. 20 AISI 1042 steel sample gradient quenched from 2100°F.

Fig. 21 Prior austenite grain size as revealed by Vilella's Etch in AISI 1042 steel sample austenitized at 1900°F, quenched and tempered at 400°F for 5 min.

Fig. 22 Vycor tube containing fettner, ductile iron and ingot iron samples for heat etch.

Fig. 23 Ductile iron sample, heat etched at 1600°F for 2 hr., showing an austenite grain size of ASTM No. 7-8.

Fig. 24 Ductile iron sample, heat etched at 1700°F for 2 hr., showing an austenite grain size of ASTM No. 7-8.

Fig. 25 Ductile iron sample, heat etched at 1800°F for 2 hr., showing an austenite grain size of ASTM No. 7.

Fig. 26 Ductile iron sample, heat etched at 1900°F for 2 hr., showing an austenite grain size of ASTM No. 6.

Fig. 27 Isothermal transformation sample revealing austenite grain size at 1600°F.

Fig. 28 Isothermal transformation sample revealing austenite grain size at 1700°F.

Fig. 29 Isothermal transformation sample revealing austenite grain size at 1800°F.

Fig. 30 Isothermal transformation sample revealing austenite grain size at 1900°F.

Fig. 31 Sample austenitized at 1700°F for 2 hr., water quenched and tempered at 1250°F for 32 hr.

Fig. 32 Sample austenitized at 1900°F for 2 hr., water quenched and tempered at 1250°F for 32 hr.
Fig. 33 Sample austenitized at 1700°F for 2 hr. and tempered at 1250°F for 5 min. .... 65
Fig. 34 Sample austenitized at 1700°F for 2 hr. and tempered at 1250°F for 10 min. .... 65
Fig. 35 Sample austenitized at 1700°F for 2 hr. and tempered at 1250°F for 25 min. .... 66
Fig. 36 Sample austenitized at 1700°F for 2 hr. and tempered at 1250°F for 30 min. .... 66
Fig. 37 Sample austenitized at 1700°F for 2 hr. and tempered at 1250°F for 2 hr. .... 67
Fig. 38 Sample austenitized at 1700°F for 2 hr. and tempered at 1000°F for 8 hr. .... 71
Fig. 39 Sample austenitized at 1700°F for 2 hr. and tempered at 1000°F for 20 hr. .... 71
Fig. 40 Sample austenitized at 1700°F for 2 hr. and tempered at 700°F for 8 hr. .... 74
Fig. 41 Sample austenitized at 1700°F for 2 hr. and tempered at 400°F for 8 hr. .... 74
Fig. 42 Sample austenitized at 1800°F for 2 hr. and air cooled .... 76
Fig. 43 Sample austenitized at 1800°F for 2 hr., air cooled and tempered at 1250°F for 5 min. .... 76
Fig. 44 Sample austenitized at 1800°F for 2 hr., air cooled and tempered at 1250°F for 30 min. .... 77
Fig. 45 Sample austenitized at 1800°F for 2 hr., air cooled and tempered at 1250°F for 2 hr. .... 77
Fig. 46 Sample austenitized at 1800°F for 2 hr. and furnace cooled .... 79
Fig. 47 Hardness variation of isothermal transformation samples at various temperatures .... 82
Fig. 48 Sample austenitized at 1800°F for 30 min. and held at 1300°F for 30 sec. .... 83
Fig. 49 Sample austenitized at 1800°F for 30 min. and held at 1300°F for 1 min. .... 85
Fig. 50 Sample austenitized at 1800°F for 30 min. and held at 1300°F for 5 min. .... 85
<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fig. 51</td>
<td>Sample austenitized at 1800°F for 30 min. and held at 1300°F for 10 min.</td>
<td>86</td>
</tr>
<tr>
<td>Fig. 52</td>
<td>Hardness variation of isothermal transformation samples at 1300°F.</td>
<td>87</td>
</tr>
<tr>
<td>Fig. 53</td>
<td>Sample austenitized at 1800°F for 30 min. and held at 1200°F for 5 sec.</td>
<td>88</td>
</tr>
<tr>
<td>Fig. 54</td>
<td>Sample austenitized at 1800°F for 30 min. and held at 1200°F for 10 sec.</td>
<td>90</td>
</tr>
<tr>
<td>Fig. 55</td>
<td>Sample austenitized at 1800°F for 30 min. and held at 1200°F for 30 sec.</td>
<td>90</td>
</tr>
<tr>
<td>Fig. 56</td>
<td>Sample austenitized at 1800°F for 30 min. and held at 1200°F for 5 min.</td>
<td>91</td>
</tr>
<tr>
<td>Fig. 57</td>
<td>Sample austenitized at 1800°F for 30 min. and held at 1200°F for 1 hr.</td>
<td>91</td>
</tr>
<tr>
<td>Fig. 58</td>
<td>Hardness variation of isothermal transformation samples at 1200°F.</td>
<td>92</td>
</tr>
<tr>
<td>Fig. 59</td>
<td>Sample austenitized at 1800°F for 30 min. and held at 1100°F for 5 sec.</td>
<td>93</td>
</tr>
<tr>
<td>Fig. 60</td>
<td>Sample austenitized at 1800°F for 30 min. and held at 1100°F for 10 sec.</td>
<td>95</td>
</tr>
<tr>
<td>Fig. 61</td>
<td>Sample austenitized at 1800°F for 30 min. and held at 1100°F for 30 sec.</td>
<td>95</td>
</tr>
<tr>
<td>Fig. 62</td>
<td>Sample austenitized at 1800°F for 30 min. and held at 1100°F for 60 sec.</td>
<td>96</td>
</tr>
<tr>
<td>Fig. 63</td>
<td>Sample austenitized at 1800°F for 30 min. and held at 1100°F for 4 hr.</td>
<td>96</td>
</tr>
<tr>
<td>Fig. 64</td>
<td>Hardness variation of isothermal transformation samples at 1100°F.</td>
<td>97</td>
</tr>
<tr>
<td>Fig. 65</td>
<td>Sample austenitized at 1800°F for 30 min. and held at 1000°F for 5 sec.</td>
<td>98</td>
</tr>
<tr>
<td>Fig. 66</td>
<td>Sample austenitized at 1800°F for 30 min. and held at 1000°F for 15 sec.</td>
<td>98</td>
</tr>
<tr>
<td>Fig. 67</td>
<td>Sample austenitized at 1800°F for 30 min. and held at 1000°F for 30 sec.</td>
<td>100</td>
</tr>
<tr>
<td>Fig. 68</td>
<td>Sample austenitized at 1800°F for 30 min. and held at 1000°F for 60 sec.</td>
<td>100</td>
</tr>
</tbody>
</table>
LIST OF TABLES

Table I. Nature of primary graphite in the core of continuous cast ductile iron bars ........................................... 10
Table II. As-quenched hardness of samples of ductile iron austenitized 2 hr. ......................................................... 17
Table III. As-quenched hardness of samples austenitized for various times .............................................................. 22
Table IV. Samples tempered at 1250°F after austenitizing 2 hr. at 1700°F and water quenched ..................................... 24
Table V. Samples tempered at 1000°F after austenitizing 2 hr. at 1700°F and water quenched .................................. 70
Table VI. Samples tempered at 700°F and 400°F after austenitizing 2 hr. at 1700°F and water quenched ..................... 73
Table VII. Hardness variation during isothermal transformation .................................................................................. 81
Table VIII. Rockwell C hardness of the samples that had been austenitized at 1550°F for 15 min. and 2 hr. and water quenched ................................................................. 116
I. INTRODUCTION

Cast iron with primary spheroidal graphite is called ductile iron. It combines the processing advantages of gray cast iron with the engineering advantages of steel. It has better properties than those of normal gray iron. Ductile and malleable irons have quite similar mechanical and other properties. However, because of the absence of a lengthy malleableizing anneal, ductile irons generally cost less than malleable iron.

Ductile iron castings are produced with a wide range of properties through the use of its excellent response to heat treatment. The original intent of this investigation was to study the effect of heat treatments on the properties of ductile iron. However, the metallographic study of the initial heat treated ductile iron samples revealed several interesting problems which were pursued in this thesis project.
II. LITERATURE REVIEW

In 1948, ductile iron was introduced to the foundry industry as a new engineering material (1)*. This material was discovered independently by the British Cast Iron Research Association and International Nickel Company. During solidification of this iron, the graphite forms spherulites instead of flakes as in ordinary gray iron.

Burkland and Rauch (2) found that the response of ductile iron to induction hardening depended on the prior microstructure. A minimum depth of 0.10 inch to RC 50 was chosen as the criterion of acceptable response to induction hardening by these authors. They found that the degree of hardening varied with the relative percentages of ferrite and pearlite in the original microstructure. As the amount of pearlite increased, both the depth of hardening and the hardness increased. That is to say, increasing amounts of combined carbon increased the response to hardening. Completely ferritized ductile iron, obtained by normalizing and tempering, hardened only to RC 51 and the depth of RC 50 was so small as to be considered nil. They concluded that combined carbon would dissolve in austenite faster than elemental carbon.

Cast iron may be somewhat similar to steel in its reaction to hardening (3). However, because of the graphite

*The figures appearing in parentheses refer to the references appended to this thesis.
and higher percentages of silicon cast irons are more difficult to austenitize than are steels. Silicon acts in two ways: 1) it promotes graphitization and graphite is much more difficult to dissolve than carbide and 2) the presence of silicon is claimed to retard the absorption of carbon by austenite. Therefore, unalloyed cast irons containing high percentages of silicon require higher austenitizing temperatures and times than steels to obtain the desired carbon content in the austenite.

Regular hardness measurements on cast irons always yield lower values than the true hardness of the metal matrix (3,4). This difference occurs because regular hardness readings average both the metallic matrix and the soft graphite. The highest hardness obtained on cast irons, RC 55 to 57, is equivalent to a matrix hardness of RC 63 to 65.

The tempering of martensite in cast irons leads to the formation of additional graphite nodules. These nodules have been called either "secondary graphite nodules" (5) or "seedling nodules" (6). The introduction of the secondary graphite nodules reduces the diffusion path for carbon and consequently may increase the rate of ferritization.

Rehder (5) and Gilbert (6) noticed the presence of secondary graphite nodules during their study of the effect of heat treatment on the mechanical properties of ductile iron. Both of these authors were studying quenching and tempering of ductile iron. By comparing mechanical
properties of two different sets of samples, one set containing primary graphite nodules in a ferrite matrix and the other containing primary and secondary graphite nodules in a ferrite matrix, Gilbert found that elongation and impact strength were low in the latter set of samples. He attributed this effect on the mechanical properties to the presence of secondary graphite nodules but he did not explain why they lowered the properties.

Gontermann (7) cited the work of Stead on the carbide phase formed in quenched iron-silicon-carbon alloys. Both these investigators were studying the ternary equilibrium diagram of the iron-silicon-carbon system. They used the method of heat tinting, that discolors the carbides, to identify the carbide phase. Stead claimed that silicon rich carbides formed during tempering of quenched iron-silicon-carbon alloys. However, the carbide should have discolored during heat tinting but when the samples were heat tinted the precipitate did not discolor. Gontermann claimed the same thing and he too was unable to positively identify the carbide phase by heat tinting. He also claimed that with increasing silicon content, more than 2.5%, and with increasing tempering temperatures, above about 550°C, the carbide phases became very unstable. However, it appears that neither of these investigators actually observed a carbide precipitate in any of their samples.

Similarities were observed in the behavior of different members of the cast iron family. Rehder (8) found that the
time-temperature relationship for the graphitization of primary cementite in a typical ductile iron was similar in nature to those found for normal white cast irons and black-heart malleable irons. He suggested that the mechanisms and kinetics of graphitization could be similar for white and ductile irons. He found that during the decomposition of pearlite in ductile iron, the carbon from the cementite precipitated on the existing graphite nodules as is the case during the annealing of malleable iron.

Loper and Takizawa (9) observed that in white cast iron the graphite spheroids, formed on malleableizing, were in contact with sulfides. The shape of the graphite formed during solidification was controlled by the temperature range over which the graphite nucleated and grew. Graphite formation in white cast iron took place during the first stage of graphitization. During this stage primary carbides decompose to form graphite. Higher first stage graphitization temperatures and increased silicon contents reduced the time required to achieve a given percent graphitization.

These authors further stated that the amount of iron sulfides in the sample's microstructure increased with lower Mn/s ratios. Higher FeS resulted in the largest number of graphite nodules and the most nearly spheroidal graphite. FeS appeared to act as nuclei for the precipitation of temper carbon.

Sandoz (10) indicated that the number of graphite nodules developed in white cast iron during malleableizing
influence the overall rate of graphitization. Furthermore, if an iron was quenched prior to the malleableizing anneal, there was a great increase in the number of nodules produced on annealing. The eutectic cementite in these quenched irons graphitized more rapidly on annealing than it did in the same irons not pretreated. He stated that this might be the result of permanent changes in the characteristics of the carbides and the austenite matrix, and such changes could alter the graphitization rate and mechanisms. He cited the work of Schwartz et. al. (11) who also stated that prequenching might change the characteristics of carbides and austenite permanently. Sandoz suggested that the changes in the characteristics of the carbides and austenite matrix might be limited to the nucleation phase of graphitization and that any influence on the nature of the cementite or austenite matrix was probably eliminated after a short time at the first stage graphitization temperature.

No literature was found on austenite grain size determination in cast irons. However, Murphy, Wood, and D'Amico (12) claimed that the prior austenite grain boundary network could be delineated by the transformation product formed during isothermal transformation of austenite in gray iron. Such a network was found in samples transformed at 1000°F.

No work on the isothermal transformation of austenite in ductile iron has been reported. Murphy, Wood, and D'Amico (12) studied the isothermal transformation of austenite in gray iron. They found that pearlite was formed in the
temperature range of $1000^\circ$ to $1300^\circ$F, acicular nonmarten-sitic products (bainite) formed in the range of $500^\circ$ to $1000^\circ$F and martensites formed below $500^\circ$F. They concluded that austenite transformation in gray iron was similar in nature to that in steel.
A. Material Studied:

This investigation was performed on continuous cast ferritic ductile iron bars. The bars were 7/8 inch in diameter and 36 inches long. Wilks (13) studied similar ferritic ductile iron bars and reported the following composition for this material:

<table>
<thead>
<tr>
<th>Element</th>
<th>Composition</th>
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<tbody>
<tr>
<td>Total Carbon</td>
<td>3.25-4.0%</td>
</tr>
<tr>
<td>Si</td>
<td>1.25-4.0%</td>
</tr>
<tr>
<td>Mn</td>
<td>0.15-0.5%</td>
</tr>
<tr>
<td>Ni</td>
<td>0.00-2.0%</td>
</tr>
<tr>
<td>Cr</td>
<td>0.30% max.</td>
</tr>
<tr>
<td>Mg</td>
<td>0.02-0.08%</td>
</tr>
<tr>
<td>Cu</td>
<td>traces</td>
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</table>

The as-received bars had variations in the size, shape and amount of graphite and in the ferrite grain size from point to point along the diameter and along the length of bar.

An area containing mainly vermicular graphite was located close to the skin of all the bars examined and it appeared to be characteristic of continuous cast ductile iron. Vermicular graphite has the shape described by Loper and Heine (14), "varying from short, stubby to more worm like". It is thicker than graphite flakes observed in an ordinary gray iron. In the core, the shape of the graphite nodules was more or less spheroidal. However, some of the
bars contained an appreciable amount of vermicular graphite in the core as shown in Table I. A number of bars were selected at random to obtain this information. The photomicrographs shown in Figure 1 and Figure 2 show core microstructures in bars that contained only spheroidal and mixed graphite shapes, respectively.

The ferrite grain size in most of the bars shown in Table I was ASTM No. 8. Two of these bars were of grain size ASTM No. 6 to 8. The coarser ferrite grains were associated with areas containing coarser nodules and the finer grain size with areas containing finer graphite nodules.

As will be shown later in the results and discussion, the as-quenched hardness of different bars sometimes varied appreciably. Apparently, the graphite nodule shape was not a factor in determining the as-quenched hardness. For example, the as-quenched hardness of samples taken from different bars with spheroidal and with mixed graphite shapes was different by one point on the Rockwell C scale. But, the as-quenched hardness of samples from different bars, austenitized at 1550°F for 2 hrs., varied from RC 11 to RC 48-49.

B. Heat Treating:

The samples were austenitized in a Thermolyne electric muffle furnace type 1700. Model No. F-A1738-1. The work chamber measured 9\(\frac{1}{2}\) inches wide, 8\(\frac{1}{2}\) inches high and 13\(\frac{1}{2}\) inches deep. The furnace was equipped with a Barber Colman
**TABLE I.**

**NATURE OF PRIMARY GRAPHITE IN THE CORE OF CONTINUOUS CAST DUCTILE IRON BARS.**

<table>
<thead>
<tr>
<th>Bar</th>
<th>Nature of Graphite*</th>
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<tr>
<td>A</td>
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<tr>
<td>B</td>
<td>S</td>
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<td>E</td>
<td>S+V</td>
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<td>I</td>
<td>S</td>
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<td>J</td>
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<td>K</td>
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<td>S+V</td>
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<tr>
<td>Z</td>
<td>S+V</td>
</tr>
</tbody>
</table>

* S  Spheroidal Primary Graphite  
V  Vermicular Primary Graphite
Figure 1: As-received sample of ductile iron which shows spheroidal primary graphite in a ferritic matrix.

Nital Etched 250 X

Figure 2: As-received sample of ductile iron which shows spheroidal and vermicular primary graphite in a ferritic matrix.

Nital Etched 250 X
controller, Model 272P capacitrol. The temperature varied about \( \pm 7.5^\circ F \) when the furnace was operated at about \( 1700^\circ F \).

Unless otherwise noted, "heating a sample to a particular temperature" means that the furnace was heated and stabilized at that temperature before the sample was placed in the furnace. Throughout the discussion, "heating time" at a particular temperature means time in the furnace which includes time for heating the sample as well as time at that temperature. The temperature was checked at regular intervals during the heat treatment using a chromel-alumel thermocouple and a Leeds and Northrup portable potentiometer, Model 8696.

All the samples were heated in air. Samples were found to have noticeable decarburization and for this reason the hardness readings and metallographic studies were carried out on sectioned samples after the cut surface was ground to remove any possible burn from the cut-off wheel.

Tempering was carried out in a smaller electric muffle furnace, type MU-55, Hevi Duty. The work chamber of this furnace measured 7\( \frac{1}{2} \) inches wide, 5 inches high and 15\( \frac{1}{2} \) inches deep. This furnace was equipped with a manually operated input controller, type J4, Automatic Temperature Control Co., to control the percentages of time during which the power was on, and a temperature controller, Model 224, Wheelco. A temperature survey for this furnace indicated that the maximum temperature variation was \( 45^\circ F \) when the furnace was operated at \( 1250^\circ F \) and the input controller set at 40%. All
the variations in temperature in both furnaces were measured at the approximate position of the samples. All the samples were air cooled after tempering.

C. Specimen Preparation:

The preparation of metallographic samples consisted of belt grinding and polishing on 1, 0, 2/0, and 3/0 grit emery papers. The final polishing was done on polishing laps using Metcloth and Elgin Dymo Diamond Compound-No. 9 universal for coarse polishing and No. 1 universal for fine polishing. Kerosene was used as a lubricant on the polishing laps. Graphite was retained by the above procedure.

D. Metallographic Studies:

Sample examination and photomicrography were carried out using a Bausch and Lomb Research Metallograph. The magnification on the ground glass screen was determined with a stage micrometer. All the samples were studied using bright field illumination. Samples were etched with either 3% or 5% nital or with picral unless otherwise noted. To identify carbides in this ductile iron, samples were boiled 5-10 minutes in alkaline sodium picrate solution as recommended by Kehl (15).

E. Hardness Tests:

Rockwell hardness tests were carried out on the quenched and tempered samples. The Rockwell C scale was used. The hardness tester was checked periodically using standard test
blocks. The matrix hardness was measured on a Kentron micro-
hardness tester using the Knoop scale with a 100 \text{ gm.} load.
IV. RESULTS AND DISCUSSION

A. Hardening of Ductile Iron:

The literature (2,16) indicates that combined carbon will dissolve in austenite faster than will graphitic carbon. Burkland and Rauch (2) found that the response of ductile iron to induction hardening depended on the prior microstructure. They noticed that the degree of hardening varied with the relative percentages of ferrite and pearlite in the original microstructure. With increasing amounts of pearlite, both the depth of hardening and the hardness increased. These authors concluded that combined carbon would dissolve in austenite more rapidly than free carbon.

A series of samples taken from two different bars of ferritic ductile iron was hardened. The samples were austenitized at various temperatures for 2 hr. and quenched in water. The two bars were selected to determine if graphite nodule shape had any effect on the response to heat treatment. Bar B had spheroidal primary graphite similar to Figure 1 while bar E had vermicular primary graphite, similar to Figure 2.

As mentioned in the literature review (3,4) regular Rockwell hardness measurements on hardened cast irons always yield lower values than the true hardness of the metal matrix. Therefore, in addition to regular Rockwell C hardness, the matrix hardness of the quenched samples was measured with a Kentron microhardness testing machine. The microhardness was measured on the Knoop scale using a 100 gm.
load. The measured microhardness was converted to the equivalent Rockwell C hardness and rounded to the nearest whole number.

Table II gives the austenitizing temperatures, the sample and the matrix hardness for the quenched samples from the two different bars. These data are shown in Figure 3. The hardness increased with increasing austenitizing temperature as expected. However, the maximum hardness of martensite, RC 65-67, for the matrix was not obtained until the samples were austenitized at 1850°F. At all temperatures below 1850°F, the hardness obtained was less than the maximum obtainable hardness.

There appeared to be no significant difference in the hardening characteristics of the two different bars with the different primary graphite nodule shapes and it was assumed that nodule shape was not a factor in determining the amount of carbon dissolved by austenite. However, while the hardness data for these two bars are in excellent agreement, the quenched hardness of other bars was found to vary very considerably. For example, if the 1550°F curve in Figure 5, to be discussed below, is extrapolated to 2 hr., it will be seen that bar would be only about RC 25 instead of RC 48-49 shown for 1550°F in Figure 3. One bar, found to be unusual in other respects, yielded RC 11 when water quenched after 2 hr. at 1550°F. The reason for these variations was not known but it was not believed to be graphite nodule shape. Presumably
### TABLE II.

**AS-QUENCHED HARDNESS OF SAMPLES OF DUCTILE IRON AUSTENITIZED 2 HR.**

<table>
<thead>
<tr>
<th>Aust. Temp., °F</th>
<th>Sample Hardness</th>
<th>Matrix Hardness</th>
<th>Equiv. RC</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Rockwell C</td>
<td>Knoop</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Bar B*</td>
<td>Bar B*</td>
<td>Bar E*</td>
</tr>
<tr>
<td>1550</td>
<td>48</td>
<td>648</td>
<td>56</td>
</tr>
<tr>
<td>1650</td>
<td>51</td>
<td>719</td>
<td>60</td>
</tr>
<tr>
<td>1750</td>
<td>53</td>
<td>794</td>
<td>63</td>
</tr>
<tr>
<td>1850</td>
<td>55</td>
<td>853</td>
<td>65</td>
</tr>
</tbody>
</table>

*Both bars were originally ferritic ductile iron with spheroidal primary graphite nodules in bar B and vermicular graphite in bar E.*
Figure 3: As-quenched hardness as a function of austenitizing temperature. All samples austenitized 2 hr.
composition variations or other unknown differences between bars account for the above hardness differences.

At all the austenitizing temperatures used in the above series the samples should have been above the eutectoid temperature and sufficient carbon should have been soluble to give the maximum hardness of RC 65-67. Apparently the austenite had not dissolved enough carbon at austenitizing temperatures below 1850°F to give the maximum hardness and the austenite may not have been saturated with carbon even at 1850°F. Figure 4 (17) shows the well known relationship between the hardness of martensite and its carbon content. Thus, as the carbon content of the martensite increases, the hardness of the martensite increases up to about 0.60% C. The maximum hardness of RC 65-67 is obtained when the carbon content of the martensite is 0.60% or more. It was found during this study that a sample hardness of RC 55-56 was equivalent to a matrix hardness of RC 65-66 in this ductile iron, which is in a good agreement with literature (3,4).

The time required to austenitize a pearlitic eutectoid steel was studied by Mehl and Roberts (18). These authors found that at 1550°F the first visible austenite, 0.5%, was formed in about 1 sec. while 99.5% austenite was present after 3 to 4 sec. Residual carbides were dissolved at this temperature in about 30 sec. and homogenization of the austenite required about 4 to 5 min. The hardness data obtained on the ductile iron samples hardened in this study indicated that the solution of the graphite in the ferritic
Figure 4: The hardness of martensite as a function of carbon content. (17).
ductile iron was very much slower than the solution of the cementite in eutectoid steel. The matrix hardness (RC 56-56) of the ductile iron samples austenitized at 1550°F indicated that the carbon content of the austenite was only 0.35% to 0.40% carbon after 2 hr. at this temperature.

Another set of samples was hardened to determine the austenitizing time required to obtain maximum hardness in the ferritic ductile iron. Samples in this study were austenitized for various times at three different temperatures, 1550°F, 1650°F, and 1800°F. The hardness data obtained on these samples are tabulated in Table III. All of bar B and most of bar E were consumed by the above study of austenitizing temperature and additional samples for this series were taken from a bar that had spheroidal primary graphite nodules. Figure 5 shows these hardness values as a function of austenitizing time for the three austenitizing temperatures.

At the lowest of the three temperatures, 1550°F, the maximum hardness of RC 55 was attained between 20 and 30 hr., at 1650°F it was attained between 3 and 4 hr. and at 1800°F between 1 and 3 hr. These results indicated that the solution of graphite in austenite is very much slower than that of cementite. However, the as-quenched hardness increased with increasing austenitizing time as was expected and indicated an increased carbon content of the austenite with prolonged austenitizing.

The hardness of the as-quenched samples did not indicate the exact amount of carbon dissolved in the austenite because,
<table>
<thead>
<tr>
<th>Aust. Time Hr.</th>
<th>Sample Hardness Rockwell C (Regular)</th>
<th>Matrix Hardness Knoop</th>
<th>Equiv. RC</th>
</tr>
</thead>
<tbody>
<tr>
<td>a. Austenitizing Temperature 1550°F</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>42</td>
<td>512</td>
<td>48</td>
</tr>
<tr>
<td>8</td>
<td>45</td>
<td>568</td>
<td>52</td>
</tr>
<tr>
<td>10</td>
<td>48</td>
<td>648</td>
<td>56</td>
</tr>
<tr>
<td>12</td>
<td>51</td>
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<td>14</td>
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<td>806</td>
<td>63</td>
</tr>
<tr>
<td>20</td>
<td>54</td>
<td>841</td>
<td>65</td>
</tr>
<tr>
<td>30</td>
<td>55</td>
<td>852</td>
<td>65</td>
</tr>
<tr>
<td>44</td>
<td>55</td>
<td>853</td>
<td>65</td>
</tr>
<tr>
<td>b. Austenitizing Temperature 1650°F</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>53</td>
<td>801</td>
<td>63</td>
</tr>
<tr>
<td>3</td>
<td>54</td>
<td>834</td>
<td>65</td>
</tr>
<tr>
<td>4</td>
<td>55</td>
<td>851</td>
<td>65</td>
</tr>
<tr>
<td>5</td>
<td>56</td>
<td>878</td>
<td>66</td>
</tr>
<tr>
<td>6</td>
<td>56</td>
<td>881</td>
<td>66</td>
</tr>
<tr>
<td>c. Austenitizing Temperature 1800°F</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>½</td>
<td>51</td>
<td>719</td>
<td>59</td>
</tr>
<tr>
<td>1</td>
<td>54</td>
<td>825</td>
<td>64</td>
</tr>
<tr>
<td>3</td>
<td>55</td>
<td>853</td>
<td>65</td>
</tr>
<tr>
<td>6</td>
<td>55</td>
<td>856</td>
<td>65</td>
</tr>
</tbody>
</table>
Figure 5: As-quenched hardness as a function of austenitizing time for three different austenitizing temperatures.
as shown in Figure 4, the hardness is the same for all austenite containing more than about 0.60% carbon. In order to study the relative amount of carbon dissolved in the austenite a set of samples was austenitized for 2 hr. at temperatures between 1500° and 1900°F, water quenched and tempered at 1250°F. It was hoped that the carbon that precipitated from the martensite during tempering, the secondary graphite, would indicate the amount of carbon dissolved in the austenite. Samples tempered for 2 hr. at 1250°F showed some clusters of very fine precipitated carbon which appeared to present a problem in this study. Therefore, other samples were tempered for 32 hr. It was found that all the clusters had coalesced after 32 hr. of tempering. The only significant difference between samples tempered for 2 hr. and for 32 hr. was the disappearance of the clusters.

An attempt was made to determine the amount of secondary graphite in the tempered samples by counting the carbon particles. However, a number of factors prevented the accumulation of meaningful data. The ductile iron was not uniform. It had appreciable variations in density of graphite nodules from one bar to the next and from point to point within each bar. This variation in the material and the fact that some of the secondary graphite particles were rather small required the examination of a large number of areas at high magnification. However, the variations in size and shape of the secondary graphite nodules within each sample and from one sample to another complicated the interpretation of graphite
nodule density measurements even if the necessary counting could be done. Counting was abandoned and the amount of precipitated carbon was evaluated qualitatively based on metallographic examination of the samples.

Essentially no secondary graphite was found in the sample austenitized at the lowest austenitizing temperature, 1500°F. This indicated that the amount of carbon dissolved in the austenite at this temperature was very small, Figure 6. Except for the 1500°F sample, the quenched and tempered samples showed a relatively large number of precipitated carbon particles. No significant change in the density of the secondary graphite nodules was observed but the size of the nodules appeared to increase with increasing austenitizing temperature. Spheroidal secondary graphite nodules were observed in the samples austenitized at 1600°F and 1700°F. The secondary graphite nodules were elongated in the 1900°F sample. A mixture of the two structures was seen in the 1800°F sample. That is, in the 1800°F sample areas were found in which the secondary graphite nodules were all spheroidal as they were in the samples austenitized at the lower temperatures while other areas had a microstructure similar to that in the 1900°F sample with elongated secondary graphite nodules, see Figures 7-10.

The metallographic examination of these samples indicated qualitatively that the amount of carbon precipitated increased with increasing austenitizing temperature. The volume of a spherical particle is a cubic function of its
Figure 6: Sample austenitized at 1500°F for 2 hr., water quenched and tempered at 1250°F for 32 hr. Sample shows equiaxed ferrite grains and essentially no secondary graphite nodules.

Nital Etched 500 X

Figure 7: Sample austenitized at 1600°F for 2 hr., water quenched and tempered at 1250°F for 32 hr. Sample shows equiaxed ferrite grains and spheroidal secondary graphite nodules.

Nital Etched 500 X
Figure 8: Sample austenitized at 1700°F for 2 hr., water quenched and tempered at 1250°F for 32 hr. Sample shows equiaxed ferrite grains and spheroidal secondary graphite nodules.

Nital Etched 500 X

Figure 9: Sample austenitized at 1800°F for 2 hr., water quenched and tempered at 1250°F for 32 hr. Sample shows some areas of equiaxed ferrite containing spheroidal secondary graphite and other areas with elongated ferrite grains containing some elongated secondary graphite nodules.

Nital Etched 500 X
Figure 10: Sample austenitized at 1900°F for 2 hr., water quenched and tempered at 1250°F for 32 hr. Sample shows elongated ferrite grains and secondary graphite nodules.

Nital Etched 500 X
diameter. Thus, a small increase in the size of the particles results in an appreciable increase in their volume and the variation in size of the graphite nodules had to be considered in evaluating the amount of precipitated carbon.

A change in the size and shape of the ferrite grains was noticed in the quenched and tempered samples, see Figures 6 to 10. At first these changes were thought to be related to the austenite grain size. A more complete discussion of this is given in the section on "Austenite Grain Size".

The effect of austenitizing time on the amount of graphite dissolved in the austenite was also studied by examining a set of tempered samples. One half of each of the samples used to study the effect of austenitizing time at 1550°F on as-quenched hardness (Table 3) was tempered at 1250°F for 2 hr. and then prepared for metallographic examination. Another set of samples was austenitized at 1900°F for times up to 2 hr. and then tempered at 1250°F for 2 hr. The microstructures of some of these samples are shown in Figures 11 to 16.

As the austenitizing time increased the amount of the carbon precipitated during tempering appeared to increase. In the 1550°F samples essentially all the secondary graphite nodules were spheroidal in shape. A large number of rather small secondary graphite particles was observed in the 1550°F samples austenitized for 6 hr. or less. These samples had
Figure 11: Sample austenitized at 1550°F for 4 hr., water quenched and tempered at 1250°F for 2 hr. Sample shows a large number of rather small spheroidal secondary graphite nodules and fine equiaxed ferrite grains of ASTM No. 7.

Nital Etched

250 X

Figure 12: Sample austenitized at 1550°F for 10 hr., water quenched and tempered at 1250°F for 2 hr. Sample shows larger spheroidal secondary graphite nodules and equiaxed ferrite grains of ASTM No. 5.

Nital Etched

250 X
Figure 13: Sample austenitized at 1550°F for 44 hr., water quenched and tempered at 1250°F for 2 hr. Sample shows large spheroidal secondary graphite and equiaxed ferrite grains of ASTM No. 5.

Nital Etched 250 X

Figure 14: Sample austenitized at 1900°F for 15 min., water quenched and tempered at 1250°F for 2 hr. Sample shows essentially all equiaxed ferrite grains, ASTM No. 7, and spheroidal secondary graphite nodules.

Nital Etched 250 X
Figure 15: Sample austenitized at 1900°F for 60 min., water quenched and tempered at 1250°F for 2 hr. Sample shows a few elongated ferrite grains and some elongated secondary graphite nodules. Most of the sample consists of equiaxed ferrite grains and spheroidal secondary graphite nodules.

Nital Etched

250 X

Figure 16: Sample austenitized at 1900°F for 2 hr., water quenched and tempered at 1250°F for 2 hr. Sample shows almost all elongated ferrite grains with elongated secondary graphite nodules.

Nital Etched

250 X
equiaxed ferrite grains of size ASTM No. 7. The microstructure of the sample austenitized for 4 hr. was typical of the samples austenitized for short times and is shown in Figure 11. All the samples austenitized for more than 6 hr. had equiaxed ferrite grains of ASTM grain size No. 5. All the samples austenitized for more than 6 hr. had about the same density of secondary graphite nodules which was less than that in the short time samples. While the density of secondary graphite nodules did not appear to change with austenitizing time the size of the nodules appeared to increase as austenitizing time increased. The reason for the abrupt change in the ferrite grain size and the density of secondary graphite nodules between the 6 and 8 hr. samples is not known.

The variation of microstructure in the samples austenitized at 1900°F was more complicated than that in the 1550°F samples. The 1900°F samples austenitized for 15 min. and less contained mostly spheroidal secondary graphite and equiaxed ferrite, Figure 14. The 2 hr. sample contained almost all elongated ferrite grains and elongated secondary graphite, Figure 16. The 30 min. and 1 hr. 1900°F samples had a mixed structure with some areas containing spheroidal secondary graphite in a matrix of equiaxed ferrite grains and other areas of elongated ferrite grains and elongated secondary graphite, Figure 15. Examination of these samples indicated that more carbon dissolved as the austenitizing time increased.
The variation in the microstructure of both the $1550^\circ F$ and $1900^\circ F$ samples just described is discussed further in the next section on "Austenite Grain Size".

B. Austenite Grain Size:

In the previous section on the hardening of ductile iron the samples austenitized at various temperatures and tempered for 32 hr. at $1250^\circ F$ (Figures 7 to 10) showed a variation in microstructure that suggested a relationship between the tempered microstructures and austenite grain size. The ferrite grains were equiaxed in the samples austenitized at $1600^\circ$ and $1700^\circ F$ and they contained spheroidal secondary graphite nodules. The sample austenitized at $1900^\circ F$ had elongated secondary graphite nodules embedded in a matrix of elongated ferrite grains. A mixed structure was observed in the sample austenitized at $1800^\circ F$. Spheroidal secondary graphite nodules were associated with areas of equiaxed ferrite grains while elongated secondary graphite nodules were found in areas of elongated ferrite grains.

The observed variation in tempered microstructure is similar to the variation of austenite grain size with austenitizing temperature in a fine grained type of steel. Typical austenite grain growth curves for a fine grained type steel are shown in Figure 17 (17). These curves show that the steel has a fine grain size at low austenitizing temperatures, a coarse grain size at high austenitizing temperatures and a mixed grain size at temperatures within the coarsening range. In the coarsening range the steel has some areas
Figure 17: Grain size as a function of austenitizing temperature for a fine-grained type steel. (17).
containing fine austenite grains and other areas of coarse austenite grains.

The 1800°F sample of ductile iron described above had mixed ferrite grain shapes and mixed secondary graphite nodules and it could have been in the corosening range of this material. Since fine grained steels, by definition, have coarsening temperatures above 1700°F, 1800°F would not be an unusual coarsening temperature. If 1800°F were the coarsening temperature of the ductile iron studied here and if the tempered microstructures were determined by the austenite grain size, the samples austenitized at temperatures below 1800°F should have had fine grained austenite and samples austenitized at 1900°F should have had coarse austenite.

The microstructures of the samples austenitized at 1550°F and 1900°F for various times (Figures 11 to 16) were studied to see if they tended to support the idea of a relationship between the austenite grain size and the tempered microstructures. These samples had been used to study the effect of austenitizing time on the amount of carbon dissolved in the austenite at 1550°F and 1900°F and were discussed above. These tempered samples could indicate the existence of a relationship between austenite grain size and tempered microstructures because there is some austenite grain growth early in the austenitizing process (19). The anticipated relationship would be indicated by equiaxed ferrite and spheroidal secondary graphite in the samples austenitized for short times while the samples austenitized for long times...
should have elongated ferrite grains and secondary graphite if coarsening of austenite occurred. A mixed structure might be found in samples austenitized for intermediate times. It was realized that the austenite might not coarsen at 1550°F even in 44 hr. and that grain growth at 1900°F might be so rapid that it would be difficult to obtain short time samples with fine austenite grain size.

Samples austenitized at 1550°F for short times, 6 hr. or less, had equiaxed ferrite grains of relatively small size, ASTM No. 7. All the samples austenitized for more than 6 hr., had equiaxed ferrite grains also but they were somewhat larger in size, ASTM No. 5, than the short time samples. The reason for the abrupt change in the ferrite grain size between the 6 and 8 hr. samples was not known. It is known that austenite grain growth in steel is relatively rapid early in the austenitizing process and that it becomes relatively slow after the first half hour (19). This ductile iron did not follow this typical steel behavior if the ferrite grain size in the tempered samples reflected a change in austenite grain size.

The samples austenitized for 6 hr. or less at 1550°F had a large number of rather small secondary graphite nodules. The samples austenitized more than 6 hr. had larger secondary graphite nodules that were fewer in number than those observed in samples austenitized for 6 hr. or less. As the time of austenitizing increased the size of secondary graphite nodules appeared to increase.
The set of samples austenitized at 1550°F neither supported nor refuted the existence of a relationship between the austenite grain size and the tempered microstructures. The sample austenitized for 44 hr. had all equiaxed ferrite grains and spheroidal secondary graphite which was similar to typical microstructures of samples austenitized at 1600° and 1700°F. The failure of this sample to indicate any coarsening of the austenite may mean that a very much longer time is required to form coarse grained austenite at 1550°F.

The samples austenitized at 1900°F tended to indicate that a relationship between the austenite grain size and tempered microstructures might exist. The short time samples austenitized for 5, 10, and 15 min. had essentially all equiaxed ferrite grains and spheroidal secondary graphite nodules. The microstructure of the sample austenitized for 15 min. was typical of the samples austenitized for short times and is shown in Figure 14. This sample had a few areas containing elongated ferrite grains. Most of these elongated ferrite grains appeared to be associated with the clusters of fine carbon particles precipitated from the martensite. As will be mentioned below in connection with the discussion of the tempering process in the ductile iron, the ferrite grains adjacent to the clusters are usually elongated and they may become equiaxed after the clusters coalesce. Since most of the ferrite was equiaxed in these short time samples, it seems reasonable to believe that the elongated grains of ferrite would become equiaxed if these samples had been
tempered longer. The clusters would disappear and the fer-
trite would become equiaxed. A cluster with elongated ferrite
grains around it can be seen about \( \frac{1}{2} \) inch above the lower
right hand corner of Figure 14.

There were a few areas in the short time samples in
which elongated grains were present but no cluster was evi-
dent. Such an area can be seen about \( \frac{1}{2} \) inch below the upper
left hand corner of Figure 14. These elongated grains might
remain elongated after prolonged tempering. If coarse
grained austenite can cause elongated ferrite grains in a
tempered sample, it is possible that this area had been
occupied by large austenite grains. Perhaps coarsening had
started in this sample.

More areas with elongated ferrite grains appeared with
additional austenitizing at 1900°F. Along with this change
in the shape of the ferrite grains, it was observed that the
shape of the secondary graphite nodules changed from a
spheroidal to a more or less elongated shape, Figures 14 to
16. The samples austenitized for 30 and 60 min. at 1900°F
had mixed microstructures while the sample austenitized for
2 hr. had essentially all elongated ferrite grains.

The samples austenitized at 1900°F for short times,
5, 10, 15 min., had equiaxed ferrite grains. These samples
could have had fine grained austenite. The samples austeni-
tized for 30 and 60 min. had mixed structures and could have
had mixed austenite grain sizes. The 2 hr. sample had essen-
tially all elongated ferrite grains and could have been
coarse grained austenite. Thus, the observed variation in the tempered microstructures of the ductile iron samples austenitized at 1900°F was the variation that would be expected if the tempered microstructure was determined by austenite grain size.

Since the samples austenitized for various times at 1900°F seemed to indicate that there could be a relationship between austenite grain size and the tempered microstructures, it was decided to confirm this relationship by determining the variation of the austenite grain size with temperature in this ductile iron and to see if the austenite actually coarsened in the vicinity of 1800°F.

When several of the standard methods used for austenite grain size determination in steels were tried on the ductile iron, it was found that austenite grain size determination in this cast iron would be a problem. Essentially nothing on austenite grain size determination in cast irons was found in the literature. Murphy, Wood, and D'Amico (12) claimed that they observed austenite grain boundaries delineated by a transformation product in a number of their samples. They were studying the isothermal transformation of gray iron. This was also observed in several isothermal transformation samples examined during this study. Two methods, heat etching and an isothermal transformation technique, were found to reveal prior austenite grain size in this ductile iron. These two methods together with the work on several of the methods that did not reveal austenite grain size are described below.
1. Gradient Quench Method (15):

In the gradient quench method about a half inch of one end of an austenitized sample about 2 or 3 inches long is quenched in water while the remainder of the sample is allowed to air cool until it is well below a red heat. One side of the sample is ground, polished, etched and examined. In such a sample there should be a great variety of structures because of different cooling rates. One or more of these structures reveal the prior austenite grain boundaries.

A machined sample of ductile iron, 2 inches long and ½ inch in diameter, was austenitized at 1750°F for 2 hr. When this sample was gradient quenched, no austenite grain boundary network was found. It was thought that a smaller sample might reveal the austenite grain boundaries. Therefore, a sample was machined down to 3/8 inch diameter and was treated as described above. This sample appeared to have a discontinuous network of transformation product. With this improvement in results, it was decided to gradient quench a still smaller sample. A 0.2 inch diameter sample also had a discontinuous network of transformation product which was essentially the same as that in the 3/8 inch sample. The transformation product was dark etching in a matrix of light etching martensite, Figure 18. The transformation product appeared to form a discontinuous network of some kind but there was some doubt about this network revealing the austenite grain size. The transformation product followed the graphite nodules to a large extent and it could have been
Figure 18: Ductile iron sample of 0.2 inch diameter that had been gradient quenched from 1750°F. The transformation product formed a discontinuous network that appears to follow the graphite nodules.

Nital Etched

100 X
nucleated by the graphite rather than by the austenite grain boundaries. The graphite nodules appeared to form a dendritic pattern, Figure 19. It did not appear that this method would reveal austenite grain size in ductile iron.

The gradient quench method was tried on an AISI 1042 steel sample to see if the procedure used was correct. The prior austenite grain boundary network as revealed near the air cooled end of the sample is shown in Figure 20. The proeutectoid ferrite nucleated at the austenite grain boundaries and delineated the prior austenite grains. A similar network was seen near the water quenched end of the sample also where the austenite grains were outlined by a dark etching transformation product in a matrix of light etching martensite.

2. Vilella's Method (15):

The austenite grain size in quenched steels can be determined by etching with Vilella's reagent. Kehl (15) claims that the best contrast is produced between the differently oriented martensite grains when the quenched sample is tempered slightly, 15 min. at 400° to 475°F.

Ductile iron samples were austenitized at 1800°F for 2 hr. and water quenched. These samples were tempered at 400°, 425°, and 475°F for various times between 5 and 30 min. Both the as-quenched and the slightly tempered samples were etched with Vilella's etch. The microstructures revealed tempered martensite but no contrast was established between
Figure 19: Ductile iron sample shown in Figure 18 in the unetched condition. The graphite nodules form a dendritic pattern.

Unetched 100 X

Figure 20: AISI 1042 steel sample gradient quenched from 2100°F. The ferrite network near air cooled end of bar reveals the location of prior austenite grain boundaries. Matrix consists of pearlite.

Nital Etched 100 X
differently oriented grains in any of the ductile iron samples.

Vilella's etch was used on quenched samples of W1 tool steel and AISI 1042 steel. A little contrast was established between martensite grains in the slightly tempered AISI 1042 steel samples, Figure 21. No martensite grain contrast was found in as-quenched or the quenched and slightly tempered samples of W1 tool steel.

3. Oxidation Method (15):

This method consists of heating a polished sample to the temperature of interest, holding and then cooling to room temperature. Heating of the sample is conducted in a mildly oxidizing atmosphere. Excessive surface oxidation is limited by packing the sample in a suitable material. If the specimen has been cooled slowly from the austenitizing temperature, repolished and etched with nital, the prior austenite grain boundaries are revealed by a network of oxide particles because oxygen penetrates most rapidly at the grain boundaries.

Polished ductile iron samples were packed in fine charcoal and heated to 1300°F for various times from ½ hr. to 4 hr. The samples were cooled slowly and repolished. No grain boundary network was revealed by a nital etch. However, the surface had been oxidized badly. This method was not studied further.
Figure 21: Prior austenite grain size as revealed by Vilella's Etch in AISI 1042 steel sample austenitized at 1900°F, quenched and tempered at 400°F for 5 min.

Etched by Vilella's Etch  100 X
4. Heat Etching (15):

Kehl (15) claims that heat etching is a simple and rapid method for revealing the austenite grain size in any type of steel, but is particularly useful for grain size determinations in steels containing less than 0.10 percent carbon. The method is based upon the fact that when a polished sample is heated into the austenite range in an inert atmosphere, the metal at the grain boundaries of the austenite is preferentially vaporized. The depressions formed at the austenite grain boundaries delineate the grains on the surface of the sample.

The method appeared to be relatively simple and rapid for ingot iron but not for ductile iron. Ingot iron samples were heat etched along with the ductile iron samples to check the procedure. At first, the polished ductile iron and ingot iron samples were sealed in a Vycor tube containing argon at 1/3 atmosphere. After heat etching it was found that the surface of the ductile iron sample was covered with a deposit of some kind. The side of the ingot iron sample adjacent to the ductile iron sample was also coated while the side farthest from the ductile iron was not. The graphite nodules on the surface of the sample were thought to be porous and, if so, they might release moisture or some other impurities during heat etching. The polished samples of ductile iron and ingot iron were soaked in acetone overnight in an attempt to extract the contaminant. Then the samples were baked in a vacuum at 200°C for 2 hr. The surfaces of these samples
showed a faint straw color indicating that the impurities were still present and it was believed that the surfaces would become oxidized or otherwise contaminated at the higher temperatures used for heat etching. These samples were repolished on the last diamond lap and were subsequently heat etched in a vacuum. After heat etching, the surfaces of these samples were indeed covered by a layer of some reaction product which obscured all details of a heat etch if one existed. This result was interpreted as evidence that the graphite nodules had adsorbed a contaminant of some kind that was volatile and that interfered with heat etching. It was decided to attempt to prepare the ductile iron samples in a manner that would not permit any contaminant except methanol and acetone to enter the graphite nodules. It was assumed that these very volatile liquids could be removed prior to sealing of the samples in the Vycor tube.

The procedure that was found to reveal the austenite grain size consisted of preliminary rough grinding on a belt grinder. Cooling water was used for this stage of preparation which was used to obtain samples of the desired size and shape. After the rough grinding, plastic gloves were worn during the remainder of sample preparation and the samples did not come in contact with any liquids except methyl alcohol and acetone. The rough ground sample was reground on a clean dry belt and polished on dry emery polishing papers. The polished samples were rinsed with alcohol and acetone and dried in air at 150°C for 2 hr.
After drying, the samples were given a light grinding on all surfaces except the polished surface on a dry belt. This removed stains, if any were present, and cleaned up the samples. Final polishing was carried out using diamond polishing compound suspended in alcohol. The polished samples were rinsed with alcohol and acetone and dried in air at 150°C for 30 min. Both the ingot iron and ductile iron samples were prepared in the same way.

One end of a Vycor tube was sealed. Chips of a Ti-Zr alloy, used as a getter, were put in the tube first. A constriction was made in the tube to separate the samples from the getter. The polished samples of ductile iron and ingot iron were transferred to the Vycor tube and the tube was evacuated using a mechanical vacuum pump. The sample tube was warmed up with a low flame of the glass blowing torch for 5-10 min. The purpose of this heating was to expel volatile matter. The samples probably did not reach temperatures appreciably above the boiling point of water. After the warming, the other end of the tube was sealed while it contained the best vacuum produced by the mechanical vacuum pump. No argon or other atmosphere was added. Figure 22 is a drawing, approximately the actual size, of a typical Vycor tube containing the samples.

The sealed sample tube was held in a vertical position so that the getter was on the top. The getter was heated to incandescence for 10-15 min. without heating the samples. Then the tube was placed in a hot furnace which had been
Figure 22: Vycor tube containing getter, ductile iron and ingot iron samples for heat etch. Approximately actual size.
heated to the desired austenitizing temperature. After 2 hr., the Vycor tube was removed from the furnace, covered with crushed insulating brick and allowed to cool to room temperature. Metallographic examination of these samples revealed the heat etches, shown in Figures 23 to 26.

The heat etched ingot iron samples showed annealing twins which are typical of a face-centered cubic structure like austenite. These annealing twins indicated that the method was revealing the austenite grain boundaries.

A gray deposit covered part of the surface of the heat etched ductile iron samples and obscured all details on the underlying metallic surface. The nature of this deposit was not investigated but it looked like it could have been carbon of some kind. Several attempts were made to improve the appearance of the heat etched ductile iron samples by polishing off this surface deposit. Whenever polishing removed the deposit it also removed the heat etch. Thus, it was not known if a heat etched surface was present under the deposit. The areas that were free of the gray deposit revealed a grain boundary network that could be used to determine austenite grain size.

The method did not reveal any austenite grain boundaries in the sample heat etched at 1500°F. It was thought that 2 hr. of heat etching might be insufficient time at this low temperature. Therefore, a sample was heat etched at 1500°F for 4 hr. but no austenite grain boundaries were found in this sample either.
Figure 23: Ductile iron sample, heat etched at 1600°F for 2 hr., showing an austenite grain size of ASTM No. 7-8.

Heat Etched 250 X

Figure 24: Ductile iron sample, heat etched at 1700°F for 2 hr., showing an austenite grain size of ASTM No. 7-8.

Heat Etched 250 X
Figure 25: Ductile iron sample, heat etched at 1800°F for 2 hr., showing an austenite grain size of ASTM No. 7.

Heat Etched 250 X

Figure 26: Ductile iron sample, heat etched at 1900°F for 2 hr., showing an austenite grain size of ASTM No. 6.

Heat Etched 250 X
The austenite grains formed at low temperatures, 1600° and 1700°F, were small. Those formed at 1800° and 1900°F were only a little larger. The grain size of the austenite was ASTM No. 7-8 at 1600° and 1700°F, No. 7 at 1800°F and No. 6 at 1900°F. At all the temperatures studied, 1600° to 1900°F, the austenite grains were relatively fine even though some grain growth was observed up to 1900°F. The type of grain coarsening displayed by the steel on which the curves in Figure 17 were obtained was not found in this ductile iron.

The first sample heat etched at 1800°F showed much larger austenite grains than that shown in Figure 25. This sample gave the impression that it had a mixed austenite grain size. Before a photomicrograph of this sample was taken its surface had deteriorated from standing. Several attempts were made to duplicate this result with other ductile iron samples but none of them reproduced this result. Figure 25 shows the microstructure of one of the duplicate samples. The austenite grains in this sample heat etched at 1800°F show evidence of grain growth. A faint network of fine austenite grain boundaries can be seen. These grain boundaries existed at some time early in the heat etching. The more prominent and larger network is believed to be the austenite grain boundaries existing at the end of 2 hr. This was more or less typical of all the bare metal areas on the sample where the heat etch could be examined.
Before discussing the significance of the austenite grain growth characteristics in the ductile iron, the results of austenite grain size determination by the isothermal transformation technique will be presented.

5. Isothermal Transformation (12):

As mentioned above, Murphy, Wood, and D'Amico (12) observed a network of transformation product in their isothermal transformation samples that delineated the austenite grain boundaries. Similar networks were observed in some of the isothermal transformation samples studied in this work. It was found possible to reveal the austenite grain size in this ductile iron by preparing suitable isothermal transformation samples.

The samples were austenitized for 30 min. at temperatures from 1500° to 1900°F. The austenitized samples were transformed at 1000°F for various times, ½, 1, 2, 3, and 5 min. One of these samples, usually the 2 min. sample in a given set, was found to have a dark etching transformation product nucleating at the austenite grain boundaries. The austenite grains themselves transformed to light etching martensite. The samples were water quenched after the predetermined transformation time and prepared for metallographic examination. The samples were broken rather than cut, mounted in a cold mount and very carefully ground to avoid heating which would temper the martensite and cause
it to etch and appear dark. The microstructures of the samples that revealed the austenite grain size are shown in Figures 27 to 30.

The results obtained by this technique were in good agreement with the results obtained by heat etching. At $1600^\circ$ and $1700^\circ$F the austenite grain size was ASTM No. 7-8, at $1800^\circ$F it was No. 6-7 and at $1900^\circ$F it was No. 6. No austenite grains were delineated in samples austenitized at $1500^\circ$F. The reason for this was not known. Several attempts to get the austenite grain size at $1500^\circ$F did not reveal any austenite grain boundaries.

6. Variation of Tempered Microstructures:

The measured austenite grain size showed that the austenite in this ductile iron did not coarsen in the vicinity of $1800^\circ$F even though some grain growth was observed up to $1900^\circ$F. In fact, this austenite did not coarsen the way it does in a fine grained type of steel as shown in Figure 17. This was believed to indicate that the variation in the tempered microstructures and the austenite grain size were not related.

The only other reasonable explanation for the variation in tempered microstructures would appear to involve the amount of carbon dissolved in the austenite and subsequently precipitated from the martensite during tempering. The austenitizing temperature and time affect both the austenite grain size and the amount of carbon taken into solution.
Figure 27: Isothermal transformation sample revealing austenite grain size at 1600°F. Sample was austenitized at 1600°F for 30 min. and transformed at 1000°F for 2 min. Bainitic network indicates prior austenite grain size of ASTM No. 7-8.

Nital Etched 500 X

Figure 28: Isothermal transformation sample revealing austenite grain size at 1700°F. Sample was austenitized at 1700°F for 30 min. and transformed at 1000°F for 2 min. Bainitic network indicates prior austenite grain size of ASTM No. 7-8.

Nital Etched 500 X
Figure 29: Isothermal transformation sample revealing austenite grain size at 1800°F. Sample was austenitized at 1800°F for 30 min. and transformed at 1000°F for 2 min. Bainitic network indicates prior austenite grain size of ASTM No. 6-7.

Nital Etched  500 X

Figure 30: Isothermal transformation sample revealing austenite grain size at 1900°F. Sample was austenitized at 1900°F for 30 min. and transformed at 1000°F for 2 min. Bainitic network indicates prior austenite grain size of ASTM No. 6.

Nital Etched  500 X
The tempering process in ductile iron is to be discussed in the next section. At this time it should be realized that early in the tempering process the carbon precipitates as very fine particles which subsequently coalesce and form secondary graphite nodules. It has been mentioned above that the fine precipitated carbon particles are present as "clusters" while coalescence is taking place and that the ferrite grains adjacent to the clusters are elongated. After the clusters coalesce and disappear the ferrite grains that were elongated become equiaxed in some samples, Figures 33 to 37. In other samples, Figures 10 and 16, apparently the ferrite does not become equiaxed. As stated above, it appears more reasonable to attribute this to increased solution of carbon by the austenite rather than to austenite grain size.

The amount of carbon dissolved in the austenite was found to increase with increasing temperature and was discussed above in the section on hardening of ductile iron. This carbon precipitated from martensite during tempering and formed secondary graphite nodules. The precipitated carbon particles were larger in the samples austenitized at 1800° and 1900°F than those in the samples austenitized at 1600° or 1700°F. It is possible that the secondary graphite particles interacted with the grain boundaries and prevented their movement. This is especially possible in the samples in which the secondary graphite was elongated.
If the ferrite grain boundaries were not free to move after the clusters coalesced during tempering, the ferrite grains might remain elongated the way they appeared adjacent to the clusters during the process of coalescing.

If the ferrite grain boundaries are pinned by large or elongated secondary graphite nodules, it should be possible to determine this by studying the location of these nodules in the various samples. The samples that had been austenitized for 2 hr. at both 1700°F and 1900°F, water quenched and tempered for 2 hr. at 1250°F were examined at very high magnification, Figures 31 and 32. These samples show that the spheroidal secondary graphite nodules in the 1700°F sample are more or less randomly distributed and are not located at the ferrite grain boundaries. On the other hand, the secondary graphite nodules in the 1900°F sample are to some extent, located in the ferrite grain boundaries and do indeed appear to be pinning these boundaries. Figure 32 also shows that the secondary graphite nodules that appeared elongated at lower magnification are probably a "string" of fine carbon particles. The distribution of the secondary graphite nodules in these tempered samples indicated that the shape of the ferrite grains was determined by the secondary graphite nodules rather than the austenite grain size.

It was assumed that the secondary graphite inhibited grain boundary movement in the samples austenitized at 1900°F and not in the samples austenitized at lower temperatures because more carbon was dissolved at the higher
Figure 31: Sample austenitized at 1700°F for 2 hr., water quenched and tempered at 1250°F for 32 hr. Sample shows randomly distributed secondary graphite.

Nital Etched

Figure 32: Sample austenitized at 1900°F for 2 hr., water quenched and tempered at 1250°F for 32 hr. Sample shows that secondary graphite has a tendency to be located on ferrite grain boundaries.

Nital Etched
austenitizing temperature. Apparently more secondary graphite was present in the 1900°F samples. It would appear that a certain minimum amount of secondary graphite might be necessary to inhibit grain boundary migration. It is possible that below a certain austenitizing temperature the solubility of carbon in the austenite is too low in this ductile iron to yield an elongated ferrite grain structure after tempering quenched samples. As shown above, samples austenitized for 44 hr. at 1550°F did not yield an elongated grain structure.

A set of samples was austenitized at 1800°F for ½, 1, 2, 6, and 24 hr. They were water quenched and tempered for 2 hr. at 1250°F. The amount of elongated ferrite grains increased with increasing austenitizing time in these samples. The 24 hr. sample contained only elongated ferrite.

On the basis of all the observations on the tempered samples it is believed that there is a minimum amount of secondary graphite required to inhibit ferrite grain boundary motion during tempering of ductile cast iron.

C. Precipitation of Carbon from Martensite:

Examination of the tempered samples during the study of hardening indicated that the carbon precipitated from martensite was always graphitic carbon when it was coarse enough to identify. Carbide was never found in these samples. It was not known whether the carbon had precipitated from the martensite as carbides and then graphitized or if it precipitated as graphite directly. The tempering process in the ductile iron was studied in order to determine the form in
which the carbon precipitated. However, this study did not answer the question. Normalized samples which are known to contain carbon were tempered to see what happened to cementite during tempering. The isothermal transformation of austenite in this material was also studied to determine the temperatures at which cementite would form. Observations made during these studies are believed to indicate that the carbon precipitated from martensite as free carbon.

1. Tempering of Quenched Ductile Iron:

a. Samples Tempered at 1250°F:

A set of samples, austenitized at 1700°F for 2 hr., was tempered at 1250°F for different lengths of time between 5 min. and 32 hr. The tempering time, hardness, and the microstructures of these tempered samples are summarized in Table IV. Some of their microstructures are shown in Figures 33 to 37 and Figure 8.

The carbon precipitated very rapidly at 1250°F. Figure 33 shows the 5 min. sample which was 100% tempered martensite. The carbon precipitated as very fine particles which began coalescing immediately. Coalescence started in the vicinity of the primary graphite nodules, Figure 34. Apparently the precipitated carbon particles went into solution and reprecipitated on the nearest primary graphite nodule leaving behind light etching ferrite areas. As tempering continued the ferritic areas grew larger, Figures 35 and 36, and isolated the uncoalesced regions into areas
## Table IV.

**Samples Tempered at 1250°F After Austenitizing 2 HR. at 1700°F and Water Quenched**

<table>
<thead>
<tr>
<th>Tempering Time</th>
<th>Hardness Rockwell C</th>
<th>Microstructure</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 min.</td>
<td>32</td>
<td>100% Tempered Martensite (Figure 33).</td>
</tr>
<tr>
<td>10 min.</td>
<td>26</td>
<td>Tempered martensite and areas with coalesced carbon particles (Figure 34).</td>
</tr>
<tr>
<td>15 min.</td>
<td>21</td>
<td>Large clusters, secondary graphite, elongated ferrite.</td>
</tr>
<tr>
<td>20 min.</td>
<td>18</td>
<td>Large clusters, secondary graphite, elongated ferrite.</td>
</tr>
<tr>
<td>25 min.</td>
<td>16</td>
<td>Clusters, secondary graphite, equiaxed and elongated ferrite. (Figure 35)</td>
</tr>
<tr>
<td>30 min.</td>
<td>15</td>
<td>Small clusters, secondary graphite, equiaxed and elongated ferrite. (Figure 36)</td>
</tr>
<tr>
<td>2 HR.</td>
<td>14</td>
<td>Very small clusters, secondary graphite and equiaxed ferrite. (Figure 37)</td>
</tr>
<tr>
<td>32 HR.</td>
<td>14</td>
<td>Secondary graphite and equiaxed ferrite (Figure 8)</td>
</tr>
</tbody>
</table>
Figure 33: Sample austenitized at 1700°F for 2 hr. and tempered at 1250°F for 5 min. Matrix is all tempered martensite.

Nital Etched 500 X

Figure 34: Sample austenitized at 1700°F for 2 hr. and tempered at 1250°F for 10 min. Coalescence of fine precipitated particles has begun in the vicinity of primary graphite nodules.

Nital Etched 500 X
Figure 35: Sample austenitized at 1700°F for 2 hr. and tempered at 1250°F for 25 min. Coalescence of precipitate into secondary graphite is well advanced. Some elongated ferrite grains can be seen adjacent to the clusters. Several unidentified particles that looked like cementite are indicated by arrows.

Nital Etched 500 X

Figure 36: Sample austenitized at 1700°F for 2 hr. and tempered at 1250°F for 30 min. Sample shows smaller clusters, secondary graphite, primary graphite and carbide like particles, several are indicated by arrows, in a matrix of ferrite.

Nital etched 500 X
Figure 37: Sample austenitized at 1700°F for 2 hr. and tempered at 1250°F for 2 hr. Coalescence of the precipitated carbon is almost complete.

Nital Etched  

500 X
which were called "clusters". Some of the precipitated particles apparently grew and these appeared to be graphite when they became large enough to identify. These coalesced particles were called "secondary graphite nodules". Eventually all the precipitated carbon coalesced and the sample contained only primary and secondary graphite in the matrix of ferrite, Figures 8 and 37.

During the process of coalescing the density of carbon particles in the periphery of the clusters was noticeably less than that in the central region of the clusters where no coalescence had occurred. The ferrite grains in the periphery of the clusters were elongated. After the carbon coalesced in these samples which had been austenitized at 1700°F the ferrite grains grew and became equiaxed. As mentioned in the previous section on austenite grain size, in cases where sufficient carbon had dissolved in the austenite, the secondary graphite particles inhibited ferrite grain boundary motion and tended to give an elongated ferrite grain structure in tempered samples.

The 25 and 30 min. samples appeared to have some carbide-like particles. Some of these particles are indicated by arrows in Figures 35 and 36. These particles displayed interfaces similar to those expected of carbides and were light etching. Two perpendicular lines were scribed on the surfaces of these samples in order to mark the location of areas containing these carbide-like particles. In this way, specific particles were examined before and after etching in
boiling sodium picrate. Since this etch darkens carbides and since the carbide-like particles did not darken it was assumed that these particles were not carbides. They could have been inclusions of some kind. The nature of these particles was not investigated further.

If any carbide had precipitated from the martensite at 1250°F it must have graphitized before the particles coalesced because no carbides were found. It was decided to temper samples at lower temperatures. If carbide precipitated from the martensite it might be possible to get it to coalesce before it graphitized.

b. Samples Tempered at 1000°F:

Another set of samples was austenitized at 1700°F for 2 hr., water quenched and tempered at 1000°F for different lengths of time between 30 min. and 20 hr. Information on these samples is tabulated in Table V.

The structural changes in the samples tempered at 1000°F were similar to those observed in the samples tempered at 1250°F. The changes were much slower at 1000°F. The 8 hr. sample, Figure 38, contained relatively large clusters. These clusters were as large as or larger than those in the sample tempered 15 min. at 1250°F. Most of the precipitated carbon had coalesced in 20 hr. at 1000°F, Figure 39, and the ferrite had become more or less equiaxed.

The 3½, 4, and 6 hr. samples had some light etching spheroidal particles that looked like carbides. As described above, lines were scribed on these samples and some of these
TABLE V.

SAMPLES TEMPERED AT 1000°F AFTER AUSTENITIZING 2 HR. AT 1700°F AND WATER QUENCHED

<table>
<thead>
<tr>
<th>Tempering Time</th>
<th>Hardness Rockwell C</th>
<th>Microstructure</th>
</tr>
</thead>
<tbody>
<tr>
<td>½ hr.</td>
<td>37</td>
<td>tempered martensite</td>
</tr>
<tr>
<td>1 hr.</td>
<td>35</td>
<td>tempered martensite</td>
</tr>
<tr>
<td>1½ hr.</td>
<td>33</td>
<td>tempered martensite</td>
</tr>
<tr>
<td>2 hr.</td>
<td>31</td>
<td>tempered martensite and areas with coalesced carbon particles.</td>
</tr>
<tr>
<td>2½ hr.</td>
<td>29</td>
<td>large clusters and areas with coalesced carbon particles.</td>
</tr>
<tr>
<td>3 hr.</td>
<td>28</td>
<td>large clusters and secondary graphite with elongated ferrite.</td>
</tr>
<tr>
<td>3½ hr.</td>
<td>27</td>
<td>large clusters and secondary graphite with elongated ferrite.</td>
</tr>
<tr>
<td>4 hr.</td>
<td>26</td>
<td>large clusters and secondary graphite with elongated ferrite.</td>
</tr>
<tr>
<td>6 hr.</td>
<td>23</td>
<td>clusters and secondary graphite with elongated ferrite.</td>
</tr>
<tr>
<td>8 hr.</td>
<td>22</td>
<td>clusters and secondary graphite with elongated ferrite.</td>
</tr>
<tr>
<td>20 hr.</td>
<td>17</td>
<td>secondary graphite, elongated and equiaxed ferrite (Figure 39).</td>
</tr>
</tbody>
</table>
Figure 38: Sample austenitized at 1700°F for 2 hr. and tempered at 1000°F for 8 hr. Sample shows large clusters and coalesced areas in which the secondary graphite nodules are fine and the ferrite tends to be elongated.

Nital Etched

Figure 39: Sample austenitized at 1700°F for 2 hr. and tempered at 1000°F for 20 hr. Primary and secondary graphite are present in a matrix of equiaxed ferrite grains.

Nital Etched
particles were examined before and after etching in boiling sodium picrate solution. This etch did not color the particles indicating that they were not carbides.

c. Samples Tempered at 700° and 400°F:

Water quenched samples, austenitized at 1700°F for 2 hr., were tempered at 400° and 700°F for different lengths of time between ½ and 8 hr., Table VI. All these samples showed tempered martensite, Figures 40 and 41. Both the microstructures and the rate of change of hardness in these 400° and 700°F tempered samples indicated that they would not coalesce in any reasonable time and these tempering temperatures were not studied further.

d. Discussion:

The study of tempering of martensite showed the changes in microstructure that occurred during tempering and it indicated that a metallographic study would not show if carbon precipitated in a free or combined state. Whenever the particles were large enough to identify in the microscope they appeared to be graphite.

It was decided to see what would happen to carbide during tempering and to see if carbide could be formed at these temperatures. This work involved tempering normalized samples and isothermal transformation studies. It is described in the following sections.
TABLE VI.

SAMPLES TEMPERED AT 700° AND 400°F AFTER AUSTENITIZING 2 HR. AT 1700°F AND
WATER QUENCHED

<table>
<thead>
<tr>
<th>Tempering Time Hr.</th>
<th>Hardness Rockwell C</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Tempered at 700°F</td>
<td>Tempered at 400°F</td>
</tr>
<tr>
<td>1/2</td>
<td>51</td>
<td>53</td>
</tr>
<tr>
<td>1</td>
<td>50</td>
<td>52</td>
</tr>
<tr>
<td>1 1/2</td>
<td>49</td>
<td>49</td>
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<td>2</td>
<td>49</td>
<td>51</td>
</tr>
<tr>
<td>2 1/2</td>
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<td>50</td>
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<td>47</td>
<td>50</td>
</tr>
<tr>
<td>8</td>
<td>47</td>
<td>49</td>
</tr>
</tbody>
</table>
Figure 40: Sample austenitized at 1700°F for 2 hr. and tempered at 700°F for 8 hr. Matrix is all tempered martensite.

Nital Etched 500 X

Figure 41: Sample austenitized at 1700°F for 2 hr. and tempered at 400°F for 8 hr. Matrix is all tempered martensite.

Nital Etched 500 X
2. Tempering of Normalized Ductile Iron:

Since tempering of quenched samples did not indicate whether the carbon from martensite precipitated in a combined or free form, it was decided to temper normalized samples which contained pearlite. It was hoped that the tempering of samples containing carbide might show what happens to carbides at the tempering temperature.

A set of samples, austenitized at 1800°F for 2 hr., was air cooled to get a pearlitic structure, Figure 42. The normalized samples displayed a typical "bull's-eye" structure. The ferrite grains formed a flower petal-like pattern around the primary graphite nodules. These normalized samples were tempered at 1250°F for 5, 10, 15, 20, 25, 30, 45, 60, 90, and 120 min. Some of the microstructures of these tempered samples are shown in Figures 43 to 45.

Tempering the normalized samples at 1250°F showed that the pearlitic structure was very unstable at this temperature. During the first five minutes of tempering there was a very noticeable increase in the amount of ferrite which indicated that the pearlite was decomposing. After about 15 or 20 min. at 1250°F the remaining pearlite appeared to be spheroidizing and this is rather apparent in the 30 min. sample shown in Figure 44. There was a small amount of spheroidized pearlite in the 90 min. sample but the 120 min. sample was completely ferritized, Figure 45.

The rather unexpected result of tempering pearlite is shown in Figure 45. After the pearlite decomposed there
Figure 42: Sample austenitized at 1800°F for 2 hr. and air cooled. Primary graphite nodules are surrounded by flower petal-like pattern of ferrite and matrix is relatively fine pearlite.

Nital Etched 500 X

Figure 43: Sample austenitized at 1800°F for 2 hr., air cooled and tempered at 1250°F for 5 min. The light etching ferrite grains around the primary graphite nodules have increased in size as a result of the decomposition of the adjacent pearlite.

Nital Etched 500 X
Figure 44: Sample austenitized at 1800°F for 2 hr., air cooled and tempered at 1250°F for 30 min. Most of the pearlite decomposed leaving ferritic areas. The remaining pearlite is at least partially spheroidized.

Nital Etched 500 X

Figure 45: Sample austenitized at 1800°F for 2 hr., air cooled and tempered at 1250°F for 2 hr. The matrix is all ferrite.

Nital Etched 500 X
were no spheroidized carbides or secondary graphite nodules. Only primary graphite nodules in a matrix of ferrite were observed in the ferritized sample. When a quenched sample was austenitized in a similar manner and then tempered at 1250°F, Figure 9, it showed a large amount of secondary graphite. It might also be mentioned here that samples that were full annealed, austenitized and furnace cooled, yielded microstructures similar to the tempered normalized samples, they contained only primary graphite nodules in a ferrite matrix, Figure 46.

The unstable nature of pearlite in this ductile iron was also observed in isothermal transformation samples. The work on isothermal transformation will be described before discussing the significance of the above observations on tempering normalized samples.

3. Isothermal Transformation of Ductile Iron:

Isothermal transformation of austenite was studied to determine the temperatures at which the carbide phase could be formed. Samples in the form of thin slices, 1/8 inch - 3/16 inch thick, were cut into two half rounds. A hole was drilled near the center of each piece. The hole served two purposes: it allowed the sample to be attached to the wire which facilitated handling during heat treatment and it allowed the samples to be broken easily. Breaking was preferred to cutting because use of an abrasive wheel tempers the martensite.
Figure 46: Sample austenitized at 1800°F for 2 hr. and furnace cooled. The matrix is all ferrite.

Nital Etched 500 X
Samples were austenitized for 30 min. at 1800°F. The austenitized samples were transformed isothermally for a measured time in a lead bath and then quenched in water. The samples were broken at the hole, mounted in a cold mount, very carefully ground to avoid heating and polished. The polished samples were etched with either nital or picral and examined.

It was not practical to follow the progress of transformation during the heat treating by polishing samples immediately. However, it was possible to measure the hardness of each sample immediately and noting the way the hardness was changing indicated the time intervals in which something was happening in the samples. With hardness change as a guide it was possible to run additional samples for the proper times to get a fairly complete picture of the course of the transformation at each of the temperatures studied. The hardness data are shown in Table VII and Figure 47. All the samples were subsequently polished and examined.

The hardness of samples quenched after 30 min. at 1800°F was found to be RC 55-56. The microstructural changes observed at each transformation temperature are described below.

a. Isothermal Transformation at 1300°F:

Transformation at 1300°F started on the surface of the primary graphite nodules with the formation of ferrite as shown in Figure 48. The ferrite started forming in less
TABLE VII.

HARDNESS VARIATION DURING ISOTHERMAL TRANSFORMATION.

SAMPLES AUSTENITIZED 30 MIN. AT 1800°F.

<table>
<thead>
<tr>
<th>Transformation Time</th>
<th>Rockwell C Hardness at Various Transformation Temperatures</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1300°F</td>
</tr>
<tr>
<td>5 sec.</td>
<td>--</td>
</tr>
<tr>
<td>10 sec.</td>
<td>--</td>
</tr>
<tr>
<td>15 sec.</td>
<td>--</td>
</tr>
<tr>
<td>30 sec.</td>
<td>49-49</td>
</tr>
<tr>
<td>45 sec.</td>
<td>48-49</td>
</tr>
<tr>
<td>1 min.</td>
<td>37-36</td>
</tr>
<tr>
<td>2 min.</td>
<td>32-30</td>
</tr>
<tr>
<td>3 min.</td>
<td>30-28</td>
</tr>
<tr>
<td>5 min.</td>
<td>26-25</td>
</tr>
<tr>
<td>10 min.</td>
<td>25-25</td>
</tr>
<tr>
<td>15 min.</td>
<td>20-19</td>
</tr>
<tr>
<td>30 min.</td>
<td>15-16</td>
</tr>
<tr>
<td>1 hr.</td>
<td>15-15</td>
</tr>
<tr>
<td>2 hr.</td>
<td>15-16</td>
</tr>
<tr>
<td>3 hr.</td>
<td>--</td>
</tr>
<tr>
<td>4 hr.</td>
<td>16-15</td>
</tr>
</tbody>
</table>

*Hardness measured at transformation time = 20 min.
Figure 47: Hardness variation of isothermal transformation samples at various temperatures.
Figure 48: Sample austenitized at 1800°F for 30 min. and held at 1300°F for 30 sec. Sample shows ferrite has started to form on the surface of some of the primary graphite nodules.

Nital Etched 250 X
than 30 sec. and continued for about 3 min. After 4 min. at 1300°F most of the austenite had transformed. The 5 min. sample contained much ferrite, a small amount of pearlite and a little martensite near the pearlitic regions, Figure 50. After 10 min. the transformation was complete and all the pearlite that had formed was decomposed. The 10 min. sample contained only primary graphite nodules in a ferrite matrix, Figure 51.

The hardness changes of samples transformed at 1300°F are shown in Figure 52. During the first minute the hardness curve dropped rapidly as a result of ferrite formation. At the start of pearlite formation the curve shows about RC 28-30, and as the pearlite formed and decomposed the hardness dropped to RC 25. Even though the sample had completed its transformation and the pearlite had decomposed during the first 10 min. at 1300°F the hardness continued to drop during the next 15-20 min. and eventually leveled off at about RC 15-16. The reason for this final hardness decrease was not known.

b. Isothermal Transformation at 1200°F:

At 1200°F the transformation was much more rapid than that at 1300°F. The sample held at 1200°F for 5 sec. showed that pearlite had already nucleated on the surface of primary graphite nodules. These pearlite colonies are indicated by arrows in Figure 53. Pearlite formation was very rapid. More than half the austenite had transformed by the end of 10 sec. and it was almost completely transformed in 30 sec.,
Figure 49: Sample austenitized at 1800°F for 30 min. and held at 1300°F for 1 min. Ferrite has grown on the primary graphite nodules.

Nital Etched

250 X

Figure 50: Sample austenitized at 1800°F for 30 min. and held at 1300°F for 5 min. This sample is mostly ferrite. There are some pearlitic areas which are associated with a small amount of martensite.

Nital Etched

250 X
Figure 51: Sample austenitized at 1800°F for 30 min. and held at 1300°F for 10 min. Matrix is all ferrite.

Nital Etched  250 X
Figure 52: Hardness variation of isothermal transformation samples at 1300°C.

Austenite transformation to free ferrite

Pearlite transformation started

Austenite transformation and pearlite decomposition completed

Rockwell C Hardness

Transformation Time

1 2 3 4 hr.

1 2 3 4 min.

1 2 3 4 sec.
Figure 53: Sample austenitized at 1800°F for 30 min. and held at 1200°F for 5 sec. The arrows indicate pearlite nucleated on the primary graphite nodules.

Picral Etched 500 X
Figures 54 and 55. There was no martensite in the 45 sec. sample which indicated that all the austenite had transformed.

The fine pearlite formed at 1200°F was very unstable. Some of the pearlite had decomposed in the 30 sec. sample as indicated by the formation of ferritic areas around the graphite nodules. Decomposition of pearlite at 1200°F was slower than at 1300°F. At 1200°F some pearlite was still present in the 1 hr. sample, Figure 57. The 2 hr. sample was completely ferritized. Spheroidization of the pearlite was noticeable after 5 min. and continued in the remaining pearlite before it decomposed, Figure 56.

The hardness of the 1200°F samples dropped rapidly to about RC 36-37 as most of the pearlite formed during the first 15 sec. The hardness remained constant at RC 36-37 up to about 3 min. when it started to drop again. This decrease in hardness was apparently associated with the decomposition of the pearlite. After about 30 min. the hardness reached its final value of RC 15-16.

c. Isothermal Transformation at 1100°F:

The structural changes in the samples transformed at 1100°F were similar to those observed in the samples transformed at 1200°F. The austenite transformation to pearlite was more rapid at 1100°F than at 1200°F but decomposition of the pearlite was slower. Pearlite formation started in less than 5 sec., Figure 59, and approximately one half of the
Figure 54: Sample austenitized at 1800°F for 30 min. and held at 1200°F for 10 sec. More than half of the austenite had transformed to pearlite.

Picral Etched 250 X

Figure 55: Sample austenitized at 1800°F for 30 min. and held at 1200°F for 30 sec. Transformation was almost complete and some pearlite near the graphite nodules had started to decompose.

Picral Etched 250 X
Figure 56: Sample austenitized at 1800°F for 30 min. and held at 1200°F for 5 min. Pearlite decomposition in the vicinity of the graphite nodules is very noticeable and the remaining pearlite is partially spheroidized.

Nital Etched

Figure 57: Sample austenitized at 1800°F for 30 min. and held at 1200°F for 1 hr. Sample shows a little undecomposed, partially spheroidized pearlite.

Picral Etched
Pearlite formation started

Pearlite decomposition started

Austenite transformation complete

Pearlite decomposition complete

Figure 5b: Hardness variation of isothermal transformation samples at 1200°F.
Figure 59: Sample austenitized at 1800°F for 30 min. and held at 1100°F for 5 sec. The arrows indicate pearlite nucleated on the primary graphite nodules.

Picral Etched 500 X
austenite had transformed in the 10 sec. sample, Figure 60. The austenite transformation was complete in approximately 30 sec., Figure 61.

The pearlite decomposed at a slower rate at 1100°F than at 1200°F. Decomposition of the pearlite started sometime between 15 and 30 sec. Light etching ferrite areas were observed in the 45 sec. sample similar to those shown adjacent to the primary graphite nodules in the 60 sec. sample in Figure 62. The pearlite had started to spheroidize after about 5 min. A little spheroidized pearlite was still present at the end of 4 hr., Figure 63.

The hardness of samples transformed at 1100°F dropped to RC 38-39 in 30 sec. while most of the pearlite was forming. The hardness remained fairly constant at RC 38-39 for about 5 min. when it started dropping again. Apparently, this drop in hardness was associated with the pearlite decomposition. The hardness drop was slower at 1100°F than at 1200°F. The hardness of the 4 hr. sample was RC 25.

d. Isothermal Transformation at 1000°F:

Transformation at 1000°F started on the surface of the primary graphite nodules with the formation of dark etching bainite. The bainite is indicated by arrows in Figure 65. The bainite also nucleated on a light etching microconstituent which appeared to be ferrite and is shown in Figure 66. The ferrite formed a network that appeared to be prior austenite grain boundaries. The ferrite started forming in less than 5 sec. and transformation of the austenite was
Figure 60: Sample austenitized at 1800°F for 30 min. and held at 1100°F for 10 sec. Approximately one half the austenite had transformed to pearlite.

Picral Etched 250 X

Figure 61: Sample austenitized at 1800°F for 30 min. and held at 1100°F for 10 sec. Pearlite decomposition at the graphite nodules is noticeable.

Picral Etched 250 X
Figure 62: Sample austenitized at 1800°F for 30 min. and held at 1100°F for 60 sec. Pearlite decomposition at the graphite nodules is noticeable.

Picral Etched 250 X

Figure 63: Sample austenitized at 1800°F for 30 min. and held at 1100°F for 4 hr. A small amount of partially spheroidized pearlite was present in this sample.

Picral Etched 250 X
Pearlite formation started

Austenite transformation completed

Spheroidization of pearlite noticed

Pearlite decomposition started

Pearlite decomposition completed in most of the areas

Figure 64: Hardness variation of isothermal transformation samples at 1100°C.
Figure 65: Sample austenitized at $1800^\circ F$ for 30 min. and held at $1000^\circ F$ for 5 sec. The arrows indicate dark etching bainite which had nucleated on the primary graphite nodules.

Nital Etched

Figure 66: Sample austenitized at $1800^\circ F$ for 30 min. and held at $1000^\circ F$ for 15 sec. The bainite nucleated on the primary graphite nodules and on the network of crystals which appear to be ferrite nucleated on the prior austenite grain boundaries.

Nital Etched
completed in about 5 min. The bainite was a feathery type. The course of austenite transformation is shown in Figures 65 to 69.

At 1000°F the bainite showed a noticeable amount of decomposition around the primary graphite nodules in the 30 min. sample, Figure 70. Some spheroidization of the bainite was observed in this sample near the primary graphite nodules. Decomposition of the bainite, indicated by a decrease in etching rate, continued slowly and was noticeable in the bainite that was still present after 4 hr., Figure 71.

The hardness changes in the samples transformed at 1000°F are shown in Figure 72. The drop in the hardness was slow during the first one half minute as a result of slow austenite to bainite transformation. After a large portion of the austenite was transformed to bainite, in about 1 min., the hardness had dropped to RC 35-36. The sharp drop in the hardness between 5 and 10 min. could have been the result of spheroidization of significant amounts of bainite. After 4 hr. the hardness was RC 26-27.

The only temperature at which a ferrite network formed was 1000°F. It was not known why this was so. At 1300°F ferrite formed on the graphite nodules prior to pearlite formation but there was no tendency to form a network at 1300°F.

e. Isothermal Transformation at 900°F:

The transformation at 900°F appeared to be different from that at other temperatures in one respect: the acicular
Figure 67: Sample austenitized at 1800°F for 30 min. and held at 1000°F for 30 sec. The sample shows feathery bainite growing on the graphite and ferrite network.

Nital Etched 500 X

Figure 68: Sample austenitized at 1800°F for 30 min. and held at 1000°F for 60 sec. Most of the austenite had transformed into feathery bainite and a ferrite network.

Nital Etched 500 X
Figure 69: Sample austenitized at 1800°F for 30 min. and held at 1000°F for 3 min. Sample shows almost complete austenite transformation.

Nital Etched

500 X

Figure 70: Sample austenitized at 1800°F for 30 min. and held at 1000°F for 30 min. Bainite adjacent to the graphite had started to decompose into ferritic areas. The bainite showed a change which appeared to be similar to spheroidization.

Nital Etched

500 X
Figure 71: Sample austenitized at 1800°F for 30 min. and held at 1000°F for 4 hr. Decomposition of the bainite is very noticeable and spheroidization has progressed.

Nital Etched 500 X
Figure 72: Hardness variation of isothermal transformation samples at 1000°F.
bainitic transformation product showed a random nucleation as well as nucleation on the surfaces of the graphite nodules. However, the bainite that appeared to be randomly nucleated may have actually nucleated on austenite grain boundaries, somewhat similar to the ferrite nucleation in the transformation at 1000°F. At 900°F austenite transformation was to an acicular bainite and was complete in about 30 min.

The bainite in the 1, 2, 3, and 4 hr. samples etched differently, more slowly, than in the sample held at 900°F for shorter times. This indicated a change in its structure and when the samples were examined in the microscope there was a subtle difference in the appearance of the bainite in the long time samples. There appeared to be some spheroidization or agglomeration of the carbide phase taking place. This gradual change in appearance of the bainite was accompanied by a slow decrease in hardness.

At 900°F there was a drop in hardness during the first minute or so as shown on the curve in Figure 78. The drop is difficult to explain. There was another significant drop between 3 min. and 30 min. when most of the austenite transformed. A further slow decrease in hardness occurred after 30 min. as the nature of the bainite changed.

4. Discussion:

The examination of quenched and tempered samples showed the presence of secondary graphite nodules. Normalized and tempered samples showed no evidence of secondary graphite
Figure 73: Sample austenitized at 1800°F for 30 min. and held at 900°F for 5 sec. Acicular bainite is indicated by arrows.

Nital Etched 500 X

Figure 74: Sample austenitized at 1800°F for 30 min. and held at 900°F for 3 min. The transformation product is dark etching, acicular bainite.

Nital Etched 500 X
Figure 75: Sample austenitized at 1800°F for 30 min. and held at 900°F for 10 min. Sample shows a matrix containing acicular bainite and a little martensite.

Nital Etched 500 X

Figure 76: Sample austenitized at 1800°F for 30 min. and held at 900°F for 30 min. Transformation to acicular bainite is essentially complete. A slight change in the etching characteristic of the bainite indicated some change in its structure.

Nital Etched 500 X
Figure 77: Sample austenitized at 1800°F for 30 min. and held at 900°F for 4 hr. Sample is still 100% bainite but the bainite had changed noticeably after its formation.

Nital Etched

500 X
Austenite transformation completed

Bainite appeared to start spheroidizing

Bainite decomposition continued

Figure 78: Hardness variation of isothermal transformation samples at 900°F.
nodules. Furthermore, isothermal transformation samples that had been held at the transformation temperature until the samples ferritized showed no secondary graphite. These observations agree with those of Burkland and Rauch (2), Rehder (5), and Gilbert (6). All these investigators found secondary graphite nodules when they examined quenched and tempered samples but observed no secondary graphite nodules in normalized and tempered ductile iron samples.

The fact that no secondary graphite nodules were formed in normalized and tempered samples and that secondary graphite nodules were observed in quenched and tempered samples, leads to the conclusion that the carbon from martensite must have precipitated directly as graphite in this ductile iron instead of precipitating as combined carbon as is the case during the tempering of steel.

As mentioned in the literature review, Gontermann (7) and Stead claimed that silicon-rich carbides precipitated during the tempering of quenched cast iron samples. However, they never actually observed carbides. It seems reasonable to believe that the fine precipitate which they assumed to be carbide was actually graphite. Their experience was apparently the same as that in this investigation. Whenever the precipitate became large enough to see, the precipitate was graphite. Gontermann and Stead assumed the carbide was very unstable and that it graphitized before it became large enough to identify.
Apparently, the nucleation of graphite is a difficult process. Graphite does not normally form in steel even though the graphite is stable relative to cementite. Appreciable amounts of silicon, which is a strong graphitizer, appear to be necessary to get graphite in cast iron. It is not difficult to obtain all combined carbon in cast iron. White irons have all their carbon in combined form and, as will be mentioned below, the nucleation of graphite in the solid state in white irons is somewhat difficult even with silicon present.

The absence of secondary graphite nodules in the normalized samples after ferritization is probably due to the difficulty of nucleating graphite. Since the cementite can go into solution easily in iron, it seems likely that the solution of the cementite, the diffusion of the carbon to the existing primary graphite nodules and the reprecipitation of the carbon on the primary graphite nodules is an easier overall process than the nucleation of secondary graphite nodules within the pearlite and the precipitation of the carbon on these nuclei. Since graphite is the stable phase, the solubility of the cementite is greater than that of graphite and a carbon concentration gradient would exist that would cause the carbon to diffuse to the graphite.

In view of the above, it seems reasonable to believe that if the carbon in the martensite precipitated as carbide this carbide would graphitize by precipitating on the primary graphite nodules just as the cementite from pearlite or
bainite does. It is difficult to believe that carbide precipitated from martensite, were this to be the case, would nucleate graphite and form secondary graphite nodules.

It is known that quenching white iron prior to the malleableizing anneal increases the number of graphite nodules (10, 11, 16). Schwartz et. al. (11) suggested that the reason for the increase in the number of nodules involved permanent changes in the characteristics of the carbides and the austenite matrix. Sandoz (10) suggested that the changes in the characteristics of the carbides and austenite might be eliminated shortly after reaching the first stage graphitization temperature and that the changes might affect only the nucleation phase of graphitization.

Both Schwartz and Sandoz implied that carbide precipitates from the martensite in the prequenched white iron and that this carbide agglomerates during heating to the first stage graphitization temperature where it graphitizes. It is not necessary to postulate a nebulous change in the characteristics of the carbide and austenite which changes the nucleation of graphite during first stage graphitization in order to explain an increase in the number of graphite nodules in prequenched white iron. A much more reasonable explanation for the increase in nodule count in prequenched iron is that the prequenching eliminates the need for graphite nucleation at the first stage graphitization temperature. During heating for the malleableizing anneal, graphite precipitates from the martensite and provides all
the graphite nuclei that can be used during first stage graphitization, see Figures 33, 40, and 41. Some agglomeration of the original graphite precipitate undoubtedly occurs during heating.

Several experiments were run to test the idea of direct precipitation of graphite from martensite. The samples that had been transformed isothermally at 900°, 1000°, and 1200°F were tempered at 1250°F for 2 hr. It was expected that on tempering the isothermal transformation samples that had been transformed partially, the areas containing martensite should be replaced by ferritic areas containing secondary graphite nodules and areas containing combined carbon, pearlite or bainite, would be replaced by areas of ferrite.

The 1200°F samples showed the anticipated microstructures after tempering the partially transformed samples, Figures 79A and 79B. Figure 79A shows the sample that had been austenitized at 1800°F for 30 min. and held at 1200°F for 10 sec. It is the same photomicrograph shown in Figure 54. In this sample the pearlite had nucleated on the primary graphite nodules and the martensite was located in areas somewhat removed from the primary graphite nodules. When this sample was tempered at 1250°F for 2 hr., the combined carbon in the pearlite apparently went into solution and reprecipitated on the existing graphite nodules. On the other hand, the carbon from the martensite formed secondary graphite. In Figure 79B it appears that two distinct types of areas correspond to the pearlite and martensite in the
Figure 79A: Sample austenitized at 1800°F for 30 min. and held at 1200°F for 10 sec. Same as Figure 54.

Picral Etched 250 X

Figure 79B: Sample austenitized at 1800°F for 30 min., held at 1200°F for 10 sec. and then tempered at 1250°F for 2 hr. Sample shows secondary graphite in areas formerly occupied by martensite and none in areas that had been pearlite.

Nital Etched 250 X
isothermally transformed sample: the completely ferritic areas correspond to the pearlitic areas and the ferritic areas containing secondary graphite nodules correspond to the martensitic areas.

In Figure 79B it can be seen that the secondary graphite nodules adjacent to the completely ferritic areas are larger than the other secondary graphite nodules. This indicates that some of the carbide from the pearlite went into solution and reprecipitated on the nearest graphite which was these secondary graphite nodules. It was apparently easier for this carbon to precipitate on these secondary graphite nodules than to diffuse to the more distant primary nodules.

The 900°C and 1000°F samples containing bainite and martensite did not display the two distinct areas, those with secondary graphite and those with none, as shown in Figure 79B. However, in all these samples the amount of secondary graphite was approximately proportional to the amount of martensite present prior to tempering. During the austenite transformation to bainite some austenite could have remained untransformed within the bainitic areas. This untransformed austenite would have formed martensite on quenching and subsequently formed secondary graphite nodules on tempering. These secondary graphite nodules were larger in size indicating that the carbon from the bainite went into solution and reprecipitated on the nearest graphite nodules.
No secondary graphite nodules were observed in the 900° and 1000°F samples that had been held at the transformation temperature for 4 hr. Apparently these samples were completely transformed to bainite.

The explanation of the precipitation of carbon from martensite was further tested by hardening a set of samples that had different original microstructures. Samples were taken from two bars. They were austenitized at 1900°F for 4 hr. Some were water quenched to get a martensitic matrix while others were air cooled to get a pearlitic matrix. The hardness of the quenched samples was RC 56 indicating that the amount of carbon in solution was 0.60% or more. The normalized samples had a microstructure similar to Figure 42 and probably contained about 0.50% combined carbon. These two kinds of samples, pearlitic and martensitic, along with as-received samples, which were ferritic, were austenitized at 1550°F and water quenched. One set was austenitized only 15 min. and a second set was austenitized for 2 hr. The hardnesses of these samples are tabulated in Table VIII.

The pearlitic, normalized samples, were the hardest and the ferritic, as-received samples, were the softest. Apparently much of the combined carbon from the pearlite went into solution in the austenite more rapidly than did the graphite. This was expected. The martensitic sample was somewhat harder than the ferritic sample which was somewhat contrary to what was expected. However, this difference is
TABLE VIII.

ROCKWELL C HARDNESS OF THE SAMPLES THAT HAD BEEN AUSTENITIZED AT 1550°F FOR 15 MIN. AND 2 HR. AND WATER QUENCHED.

<table>
<thead>
<tr>
<th>Austenitizing Time at 1550°F</th>
<th>Original Condition of Samples</th>
<th>Austenitized at 1900°F for 4 hr. and water quenched (Martensite)</th>
<th>Austenitized at 1900°F for 4 hr. and air cooled (Pearlite and some ferrite)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>As-received (Ferrite)</td>
<td>Bar Y</td>
<td>Bar Z</td>
</tr>
<tr>
<td>15 min.</td>
<td>16</td>
<td>16</td>
<td>25</td>
</tr>
<tr>
<td>2 hr.</td>
<td>38</td>
<td>37</td>
<td>42</td>
</tr>
</tbody>
</table>
reasonable because the martensitic sample probably precipitated secondary graphite during heating and this graphite dispersion, being much finer than the primary graphite in the ferritic sample, would dissolve somewhat faster yielding a higher carbon austenite and harder martensite. Thus, the variation in hardness of the three kinds of quenched samples is the variation to be expected if carbon precipitates from martensite as graphite in cast irons.

The hardness data shown in Table VIII show that the difference between the three kinds of samples was more pronounced with the short, 15 min., austenitizing than it was with the 2 hr. austenitizing. This should be the case since the combined carbon should go into solution very rapidly and the longer time would allow the graphite "to catch up" with the solution of the combined carbon. It should be mentioned that the 15 min. ferritic sample did not give a completely martensitic sample after quenching while the other two kinds of samples were completely martensitic.

The reason for the direct precipitation of graphite from martensite in cast irons is not known. However, it seems reasonable to believe that carbon does precipitate as graphite rather than as carbide as is the case in steels.

D. Repeated Quenching and Tempering:

If there is a difference between the primary and secondary graphite nodules in ductile iron, it should be possible to change from a dispersion of primary graphite nodules to a dispersion of secondary graphite nodules by repeated quenching
and tempering. In other words, after repeated quenching and tempering it may be possible to dissolve the primary graphite nodules and to allow the secondary graphite nodules to grow and finally to achieve a more or less uniform graphite size. A change in graphite nodule dispersion could have a significant effect on the mechanical properties. Therefore, an attempt was made to change the graphite dispersion by repeated quenching and tempering.

Samples were austenitized at $1800^\circ F$ for 2 hr., water quenched and tempered at $1250^\circ F$ for 2 hr. Nine different samples that had been given 1, 2, 3, 4, 6, 9, 12, 15, and 21 of these heat treating cycles were examined. Figures 80, 81, and 82 show the microstructures of samples treated for 4, 12, and 21 cycles.

In Figures 80, 81, and 82 there appears to be an increase in the amount of primary graphite with increasing number of cycles. These microstructures were obtained from three different samples and the variation of primary graphite should be attributed to the nonuniform nature of this ductile iron rather than to the number of heat treating cycles. The variation in the secondary graphite and in the ferrite grain size can be attributed to the number of heat treating cycles.

There was some increase in the size and amount of secondary graphite nodules with increasing number of cycles. The secondary graphite nodules tended to be less dense
Figure 80: Ductile iron sample repeated quenched and tempered 4 times. Sample was austenitized at 1800°F for 2 hr., water quenched and tempered at 1250°F for 2 hr.

Nital Etched

250 X

Figure 81: Ductile iron sample repeated quenched and tempered 12 times. Sample was austenitized at 1800°F for 2 hr., water quenched and tempered at 1250°F for 2 hr.

Nital Etched

250 X
Figure 82: Ductile iron sample repeated quenched and tempered 21 times. Sample was austenitized at 1800°F for 2 hr., water quenched and tempered at 1250°F for 2 hr.

Nital Etched 250 X
immediately adjacent to the primary graphite nodules indicating that most of the carbon from these areas had precipitated on the primary graphite nodules.

As the amount of secondary graphite increased in regions between the primary graphite nodules it affected the ferrite grain size. The presence of sufficient secondary graphite inhibited grain growth in the ferrite and it will be seen in the photomicrographs that the ferrite grain size is much finer in the areas containing secondary graphite nodules. Furthermore, the ferrite grain size is somewhat smaller in the samples which were quenched and tempered a greater number of times.

It would appear that it might be possible to achieve a more or less uniform distribution of secondary graphite nodules and to eliminate the original dispersion of primary graphite nodules by repeated quenching and tempering. However, it would appear that a very large number of heat treating cycles would be required to accomplish this.

E. Relationship Between the Hardness Readings:

As described in the section on hardening of ductile iron, the sample hardness of quenched ductile iron was lower than the true hardness of the metal matrix. All the regular Rockwell C hardness readings were plotted against the equivalent Rockwell C hardness of the matrix as shown in Figure 83. The matrix hardness had been measured with the Kentron microhardness testing machine on the Knoop scale using a 100 gm. load. It appears that the relationship between the two
Figure 83: Relationship between sample hardness and matrix hardness.
hardness values is linear and can be represented by the equation:

\[ M = 1.4 \times S - 11.0 \]

Where, \( M \) is the matrix hardness, Rockwell C converted from Knoop,

\( S \) is the sample hardness, measured Rockwell C.

The highest sample hardness attained in cast irons, RC 55 to 57, was equivalent to a matrix hardness of HC 63 to 65 and was in a good agreement with the literature (3,4).

F. Isothermal Transformation Diagram:

Information obtained on this ductile cast iron permits drawing a partial transformation diagram. This diagram based on hardness data and metallographic observations is approximate and is shown in Figure 54. The diagram is somewhat similar to that reported for gray iron by Murphy, Wood, and D'Amico (12).

The product of austenite transformation was found to be unstable at each of the temperatures studied. Dashed lines are shown on the diagram to indicate the approximate times at which the transformation product started to decompose and at which decomposition was complete.
Figure 84: Isothermal transformation diagram for continuous cast ductile iron.
V. CONCLUSIONS

From the results and observations made during the course of this investigation the following conclusions have been reached:

1. The solution of graphite in ductile iron was much slower than the solution of carbides. This material requires a long austenitizing time at a high austenitizing temperature for full hardening.

2. Austenite grain size determination in ductile iron was more difficult than in steel. Only two methods, heat etching and an isothermal transformation technique, revealed the prior austenite grain size in the ductile iron.

3. When quenched ductile iron samples were tempered at 1250°F until ferritization was complete, the final microstructure varied with the prior austenitizing treatment. The amount of carbon dissolved in the austenite and subsequently precipitated from the martensite is believed to determine the shape of the final ferrite grains and the distribution of the secondary graphite. Smaller amounts of dissolved carbon result in a random dispersion of secondary graphite in a matrix of equiaxed ferrite grains. Larger amounts of dissolved carbon yielded an elongated ferrite grain structure with strings of fine secondary graphite nodules in the grain boundaries and some randomly distributed secondary graphite.
4. Secondary graphite nodules were formed only when ductile iron samples containing martensite were tempered. It is believed that the carbon from martensite precipitated directly as graphite rather than as combined carbon as is the case with steels. The reason for direct precipitation of graphite from martensite is not known. When samples containing combined carbon but no martensite were tempered no secondary graphite was formed. Apparently, the combined carbon went into solution and reprecipitated on existing graphite nodules as is the case during annealing of black heart malleable irons.

5. It might be possible to change the primary graphite dispersion into a secondary graphite dispersion by repeated quenching and tempering. This might have a significant effect on the mechanical properties. It appeared that a very large number of cycles would be required to change the graphite dispersion and repeated quenching and tempering does not appear to be a process with practical value.

6. The regular Rockwell C and the matrix hardness were related by the relationship:

Matrix hardness = 1.4 x Sample hardness - 11.

This agrees well with a similar relationship found in the literature.

7. A part of the isothermal transformation diagram for ductile iron was determined and appears to be similar to the diagram for ordinary gray iron.


VITA

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