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SPHEROIDIZATION IN TOOL STEELS

BY

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THESIS

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Approved by

Fred Kisslinger (advisor) Xavier JR Avula

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ABSTRACT

A carbon steel, W1, and four other alloy tool steels, namely, A2, H13, D3, and M2, were spheroidized by five different procedures. Any modification made in the treatments for an individual steel were suggested by the results obtained with these five procedures. The hardnesses and microstructures obtained were compared with steel samples annealed at a tool steel mill. The response given by different steels to various methods of spheroidization was studied. It was found that A2, W1, and D3 steels responded the best to the cycling method whereas the best response to the isothermal annealing was given by H13 and M2 steels. The effect of neutron irradiation on the spheroidization of W1 steel was studied and it was concluded that the radiation damage in W1 steel did not affect the rate of spheroidization.

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The author is also indebted to Columbia Tool Steel Company for their generosity in supplying the steels used in this study.

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NOMENCLATURE

Dia. : Diameter

Hr : Hour(s)

P.S.P.: Partially spheroidized pearlite

RB : Rockwell B hardness

RC : Rockwell C hardness

T_a : Austenitizing temperature

t_a : Austenitizing time

Temp. : Temperature

T_n : Upper nose temperature

t_n : Time at the upper nose temperature

T_s : Spheroidizing temperature

t_s : Spheroidizing time

I. INTRODUCTION

Spheroidization is a process of heating and cooling steel that produces a rounded or globular form of carbide. A spheroidized structure may be desired for a number of reasons, the two principal reasons being to facilitate machining certain parts and to obtain uniformity in hardening some grades of steel. The modern tendency to displace hot forging and, to some extent, machining, by cold forming in dies under tremendous pressure - cold extrusion - has intensified the need to produce very soft, highly spheroidized structures in carbon and alloy machinery steels.

Spheroidizing is done in various ways, depending on the prior structure and nature and composition of the steel being treated. There are several factors that affect the rate of spheroidization and a judicious choice of one or a combination of these should be used to obtain a given rate of spheroidization. However, with current trends in industry geared to obtain maximum productivity of equipment, the faster the rate of spheroidization, the cheaper will be the process involving it. It is with this intention that an attempt has been made to use a number of variables effectively so as to get an optimum spheroidized structure at the fastest rate in various tool steels.

II. LITERATURE REVIEW

A. General:

Spheroidization has been defined in a report(1)* of Committee E-8 of the American Society for Testing Materials as a process of heating and cooling steel that produces a rounded or globular form of carbide.

The driving force of spheroidization is the reduction in surface energy of the carbide. It is possible to spheroidize merely by holding any steel at a temperature below the lower critical temperature.

The process of spheroidization in steel is utilized mainly for high carbon and alloy steels. The primary purpose of this process is to facilitate machining of these steels thereby reducing the cost of production of machined parts and increasing tool life.

The term annealing covers the heating of steel to temperatures below the critical as well as to temperatures above the critical. The former is quite simple and straightforward since no metallurgical phase changes occur below the critical temperature. The latter depends almost entirely on two factors, namely, the formation of austenite and the subsequent transformation of austenite at temperatures just below the critical range. The more carefully these factors are controlled, the more successful

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*Numbers in parenthesis refer to bibliographical entries.

is the operation. The product of annealing is governed by three main rules as given by Peter Payson(2):

> Rule 1: The more homogeneous the structure of the steel as austenitized, the more completely lamellar will be the carbide in the annealed steel. Conversely, the more heterogeneous the structure of the steel as austenitized, the more completely spheroidal will be the carbide in the annealed steel. Consequently, the higher the austenitizing temperature, the greater is the tendency for the structure of the annealed steel to be lamellar whereas the closer the austenitizing temperature is to the critical range, the greater is the tendency for the structure of the annealed steel to be spheroidal.
>
> Rule 2: The softest condition of a steel is developed by austenitizing at a temperature usually less than 100^oF above the critical range, and transforming at a temperature usually less than 100^oF below the critical range.
>
> Rule 3: Since the time for complete transformation at temperatures less than 100^oF below the critical range may be very long, most of the transformation should be allowed to take place at the higher temperature where a soft product is formed and

the transformation should be finished at a lower temperature where the time for the complete transformation is short.

These rules apply particularly to carbon steels. In fact, most of the discussion in this chapter strictly applies to low alloy and carbon steels. Spheroidization in highly alloyed tool steels has not been discussed extensively in the literature. The subject matter of this thesis is spheroidization in these highly alloyed tool steels. Most of these steels do not form pearlite. However, an attempt has been made to investigate how the rules and the discussion in this chapter apply to the highly alloyed tool steels.

Based on Payson's Rules, various methods of spheroidization(3) are available. They are as follows:

i) Prolonged holding at a temperature just below the lower critical temperature.

ii) Cycling method - heating and cooling alternately between temperatures that are above and below the lower critical temperature.

iii) Isothermal annealing - austenitizing the steel and allowing it to transform isothermally just below the lower critical temperature. A very similar process involves very slow cooling from the austenitizing temperature.

iv) Cooling at a suitable rate from the minimum temperature at which all carbide is dissolved to prevent the reformation of a carbide network and then reheating in accordance with any of the above methods. (Applicable to hypereutectoid steels containing a carbide network. Not applicable to highly alloyed tool steels in which all the carbide cannot be put into solution at temperatures below the solidus temperature.)

While spheroidization of cementite occurs, the grain size of the ferrite increases and this has been correlated with the change in mechanical properties by Hyam and Nutting(4). The ability of cementite to spheroidize is attributed to its complex crystal structure, orthorhombic, which makes it almost isotropic.

The driving force for spheroidization is believed to be associated with the reduction in interfacial area between the cementite and the ferrite matrix. The carbon content of the ferrite in equilibrium with the cementite plates will be higher at the ends of the plates where the radius of curvature of the interface is smaller than at the flat sides where the radius of curvature is relatively large. The process of spheroidization occurs by the simultaneous solution, diffusion through the matrix and deposition of the cementite. Several workers investigated the thermodynamics of this process and came to the conclusion that is summarized by Hyam and Nutting, that the activation energy of the softening process is of the order of 60 kcal per g-mole. These authors also mentioned that although the quantity varies with the actual initial hardness, this value gives a fairly good mean taken over the range of hardnesses generally encountered.

Hyam and Nutting have further extended a possible mechanism for this process, based on a discussion by Cottrell and Leak(5) who reasoned that since the solubility of small carbide particles is greater than that of the larger carbide particles, the small carbides will go into solution and the carbon will diffuse to the larger carbides where it reprecipitates. In this way, the larger carbides grow larger while the smaller carbides disappear.

If this mechanism alone accounted for the change in carbide particle size, then, according to Hyam and Nutting the rate controlling process would be the diffusion of carbon in ferrite, the activation energy of which is known to be 20 kcal per g-mole. The activation energy of spheroidization is known to vary from about 12 kcal per g-mole in the initial stages(6,7) to about 60 kcal per g-mole in the later stages. This could indicate that the initial and final stages have different mechanisms. The high activation energy required in the final stages may be explained as follows. The final stages of spheroidization involve the growth of the larger carbide particles at the expense of the smaller ones. For smaller carbides to disappear, the carbon from these particles must go into solution to form ferrite. This change from cementite to ferrite involves an increase in density, and thus the net effect of transferring iron atoms from cementite to ferrite lattice positions will be the production of vacancies. These vacancies will have to diffuse away before more of the cementite particles can go into solution. Similarly, vacancies must diffuse to and be annihilated at the growing cementite particles. The movement of vacancies may, therefore, be the rate controlling process and this would require an activation energy of self-diffusion. This is in contrast to the low values of activation energy found for the early stages when the majority of carbide particles are coherent with the matrix and are growing larger by depleting the surrounding matrix of carbon.

It will always be noticed that none of the cementite particles are perfectly circular or even ellipsoidal at the sectioning plane of observation. The reason for this lies in the amount of energy required to be expended in creating different portions of the particle-matrix interface. Different positions will mean contact of the cementite with different planes of the ferrite lattice. Thus, the interfacial energy is not the same at all points on the interface resulting in the distortion of the particle from a perfectly spherical or spheroidal shape to the unsymmetrical shapes that are seen under the microscope.

The discussion up to this point was concerned with the mechanism and general nature of the spheroidization process. Various factors that affect the rate of spheroidization will now be discussed.

B. Variables Affecting the Rate of Spheroidization:

1. Temperature: Of all possible aggregates of ferrite and cementite, the spheroidized structure is, by far, the most stable. For this reason, merely by holding a steel at a suitable temperature below Ae_1, spheroidization will occur at a fair rate. The higher the temperature, the faster will be the rate of spheroidization, since the rate of diffusion increases with increasing temperature.

2. Original Microstructure: As mentioned earlier, spheroidite* can be obtained from any initial microstructure; the only point of difference is the relative ease of spheroidization. Spheroidite can be obtained from either pearlite or bainite or martensite by holding for a suitable time just below the eutectoid temperature. A general rule(8) that may be formulated in this respect is that the finer the initial dispersion of the carbide, the faster will be the rate of spheroidization. From this, it follows that the rate will be slowest when the starting structure is coarse pearlite(8,9) and it is also true that the coarser the pearlite the more difficult is spheroidization. From the same argument, it follows that bainite is easier to spheroidize and tempered martensite is the easiest. An additional reason for the ease of spheroidizing a martensitic structure is that martensite is a highly unstable phase and moves readily in the direction of stable equilibrium when tempered. The

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*Coarsely spheroidized cementite dispersed in a ferrite matrix.

excess carbon that is trapped in the body-centered tetragonal lattice of martensite is precipitated as cementite and the carbon content of the martensite falls rapidly. When cementite just forms, it appears as a film(10) at the boundaries of the martensite plates, and as globules and platelets inside the martensite plates. As tempering proceeds, these particles grow in size and tend to achieve a spheroidal shape.

3. Prior Cold Working: It is a well-known fact that cold working prior to a spheroidizing treatment enhances the rate of spheroidization. This can be attributed to a variety of defects produced by cold working. Vacancies provide an easy path for the diffusion of carbon; however, diffusion along grain boundaries and other short circuiting paths is also known to exist.

Holtzman et al(11) have studied the spheroidization of cold worked pearlite. By using electron microscope techniques, they found that deformation takes place by

i) ferrite slip which is parallel to the lamellae,
ii) slip transverse to the lamellae with both cementite and ferrite deforming, and
iii) ferrite-cementite interfacial slip.

The transverse slip appears as a "stepped" area when examined with a light-microscope. The result of the investigation was the confirmation of the statement that spheroidization begins in these "stepped" areas.

It was seen that the lamellae form a step by bending at two points along their length and a "step band" is generated across the pearlite colonies. The microstructures presented in the paper showed that spheroidization started in these "step bands". This was in sharp contrast to the spheroidization that occurred in undeformed pearlite where the carbide lamallae "pinch off" at more or less regular intervals to form small ellipsoidal carbides which subsequently spheroidize. Thus, spheroidization in undeformed pearlite occurred almost randomly throughout the colony.

In deformed pearlite, the step bands increase in severity and number with increasing deformation and, as a result, more carbide regions are available for inducing spheroidization. The rate of spheroidization is, therefore, considerably enhanced by increasing the deformation.

4. Concurrent Deformation: This factor is, by far, the most influencial in accelerating the rate of spheroidization in steels. Robins et al(12) have shown that at a concurrent strain rate of 2.4 per cent per second, full spheroidization in a commercial grade eutectoid steel can be made to occur in less than three minutes at 700°C. This phenomenom has been explained by them on the following grounds:

i) Concurrent hot working during spheroidization produces lattice vacancies, as in the case of cold working. These excess point defects accelerate the mobility of atoms and contribute to an enhancement in the rate of spheroidization.

ii) Another possible explanation is based on the self diffusion of iron. It is well known that concurrent plastic deformation enhances the self-diffusivity of ferrite at high temperatures (700°C - 900°C). The diffusivity increases with increasing strain rate, but this dependence decreases with increasing temperature.

iii) Another mechanism that may be used to explain this phenomenon is based on the net increase in the grain boundary area, brought about by working above recrystallization temperature. This may provide short circuiting paths for diffusion of iron and carbon.

5. <u>Creation of Vacancies</u>: The cycling method of spheroidization (Method 2, pg29) involves some amount of phase transformation of the steel. Since a change in volume is associated with this transformation, it is possible that point defects are created, which result in an acceleration in the rate of spheroidization.

6. <u>Alloy Content</u>: The precipitation of carbides in alloy steels exhibits a unique feature during high temperature tempering (a spheroidization process), in that the carbide-forming elements concentrate in the carbides with increasing tempering temperatures. The carbide-forming elements that may be present in a steel diffuse into the carbide that forms initially. When sufficiently enriched, this carbide

transforms to the higher alloy carbides. After further enrichment this carbide may be superseded by another and this formation of transition carbides may be repeated several times before the equilibrium carbide forms. Crafts and Lamont(13) have shown, in carbides formed at tempering temperatures above 1000°F, enrichment of manganese in the case of a manganese steel, and enrichment of chromium in the case of chromium steel. This enrichment occurs in chromium steels in the following stages: Fe_3C to Cr_7C_3 to $Cr_{23}C_6$. In such steels, therefore, a structure containing cementite is not the most stable structure possible and it is not the end product of prolonged tempering.

For spheroidization to occur during isothermal annealing of straight carbon steel, only carbon has to diffuse, since the matrix contains iron. In isothermal annealing of alloy steels, however, spheroidized carbides form by simultaneous diffusion of carbon and carbide forming elements. These alloying elements have high atomic numbers and low diffusion coefficients. Because of this, in general, the alloying elements retard the rate of spheroidization.

Smith(14) studied the effect of aluminum and silicon deoxidizers which are used to control the grain growth characteristics of austenite. He claimed a slight but definite tendency for spheroidization to proceed at a more rapid rate in steels deoxidized with aluminum than those deoxidized with silicon.

III. EXPERIMENTAL PROCEDURE AND RESULTS

A. General:

1. Material: The steels studied in this investigation were received from the mill in the hot-rolled air-cooled condition. They were in the form of $\frac{1}{2}$ inch rounds. Their compositions and as-received hardnesses are given in Table I.

2. Furnace Control: All the samples were heated in air. Unless otherwise specified, "heating a sample to a particular temperature" implies that the furnace was heated and stabilized at that temperature before the sample was placed in it. Temperature was checked with a chromel-alumel thermocouple and a Leeds & Northrup Model 8696 potentiometer. Temperature was controlled to $\pm 5^{\circ}F$. Only one furnace was used for all the operations involving furnace cooling of a sample. The rate of cooling up to about $1000^{\circ}F$ in this furnace is given by the cooling curve in Fig. 1. From the curve, it is clear that the temperature was about $1000^{\circ}F$ after cooling for 3 hours from $1800^{\circ}F$. It was also observed that the temperature was about $700^{\circ}F$ at the end of the fifth hour, about $300^{\circ}F$ at the end of the tenth hour, and about $100^{\circ}F$ at the end of the fifteenth hour.

3. Hardness Testing: Hardness testing was done on the B and C scales of a Rockwell hardness testing machine. Before testing hardness, the decarburized layer on the surface was completely ground off. Reproducibility

TABLE I.

Compositions and As-received Hardnesses of the Steels Studied

Steel	C	Mn	Si	W	Mo	Cr	V	As-received hardness, RC
A2	1.01	0.57	0.28	---	0.98	5.12	0.25	44
D3	2.12	0.31	0.98	0.66	0.09	11.55	---	48
H13	0.38	0.29	0.74	---	1.34	5.29	1.01	52
M2	0.85	0.33	0.36	6.18	4.76	3.96	1.83	57
W1*	1.07	0.28	0.27	---	---	0.06	---	30

*The manufacturer calls this steel W1-2.

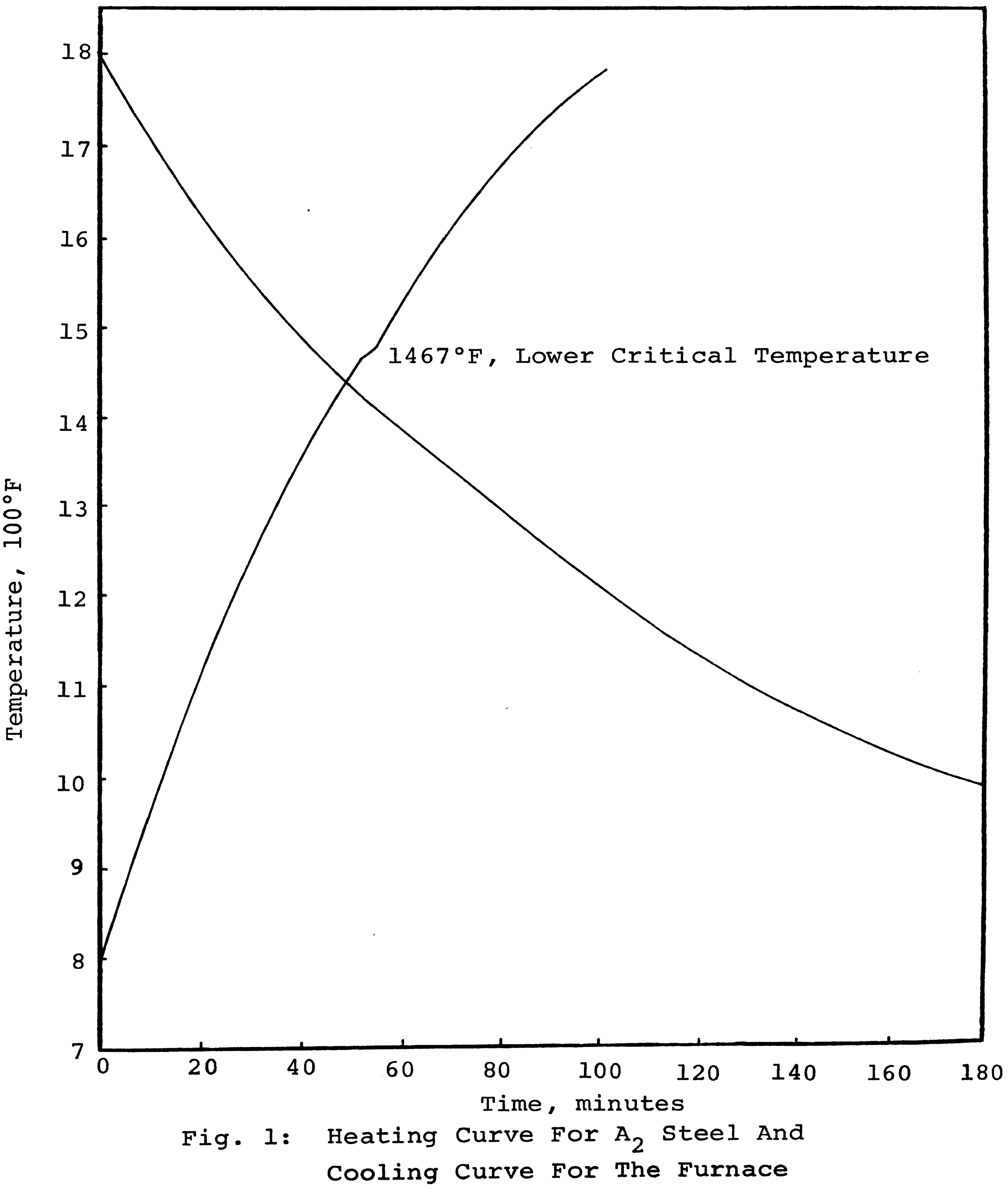

Fig. 1: Heating Curve For A_2 Steel And Cooling Curve For The Furnace

of the hardness values was checked by taking two or more readings, which usually agreed within about one point.

4. Metallographic Techniques: Final polishing of all the samples was done with the help of diamond abrasives. 5% and 10% nital solutions were used as etching reagents. All the samples shown in the photomicrographs were etched with a 5% nital solution and were photographed at a magnification of 1000X.

B. Determination of Lower Critical Temperatures:

1. Thermal Analysis: This determination was made by the simultaneous "thermal analysis" of three different steels. When a steel is heated a decalescence occurs on passing through the critical temperature. This decalescence is associated with the absorption of heat which occurs when the ferrite-carbide aggregate changes to austenite. This fact was used for the determination of the lower critical temperatures of the steels.

Pieces about 2" long were cut from each of the three different steel bars. A longitudinal hole about 1/8" in diameter and 1" deep was drilled at the center of each piece. To facilitate drilling, each piece was softened to about 25-30 Rockwell C by tempering. Thermocouples were inserted in the holes of the three pieces so as to allow the tips of the thermocouples to touch the metal. These three thermocouples were connected to a single potentiometer using a selector switch system. The three pieces were then placed in a cold furnace. The furnace was turned on and the rate of heating was adjusted to a low value. The temperature of each piece was measured every minute (the potentiometer was read every 20 seconds) until the temperature exceeded the lower critical temperature. The power to the furnace was then turned off and again temperature readings were taken every minute during cooling.

The heating curves for each of the five steels studied showed an arrest at the lower critical temperature. However, because of the high hardenabilities of these steels, none of the alloy steels showed the lower critical temperature during cooling. Typical of the heating curves is that for A2 steel shown in Fig. 1. Table II gives the lower critical temperatures of the different steels. Wherever possible they are compared with values obtained from the literature.

The sluggish nature of the phase transformations in many of these tool steels made the reliability of the thermal analysis questionable. For this reason, the results were checked by tempering hardened samples to determine where austenite started to form.

2. <u>Heating and Quenching of A2 Steel</u>: Seven as-received samples of A2 steel were preheated at 1450°F for 30 minutes and then heated to 1700°F for an additional 30 minutes before they were oil quenched. The as-quenched hardness was 61 RC. These hardened samples were then tempered for 4 hr at 400°F, 600°F, 800°F, 1000°F, 1200°F, 1400°F, and 1500°F. The

TABLE II.

Lower Critical Temperatures of the Steels Studied

Steel	Lower Critical Temperature, °F			Ts, °F
	thermal analysis	heating & quenching	literature	
A2	1467	between 1400 & 1500	1460	1425
H13	1564	between 1500 & 1600	1560	1525
D3	1490	between 1475 & 1500	----	1460
M2	1500	between 1475 & 1500	1500	1475
W1-2	1346	between 1325 & 1350	1350	1325

samples were then water quenched from their tempering temperatures. The hardness of each sample was measured after tempering. These hardness values are given in Table III.

As long as the tempering temperature is below the lower critical, there should be a decrease in hardness with increasing temperature. But, when the temperature exceeds the lower critical, austenite forms and transforms to martensite on quenching.

The increase in hardness at 1500°F indicated that the critical temperature was between 1400°F and 1500°F. Thus, the value of the critical temperature found earlier, 1467°F, appeared to be reasonable. Furthermore, it agreed with the value in the literature, 1460°F(15).

H13, D3, M2, and W1 steels were studied somewhat differently from A2 steel. As-received samples of these steels were not hardened. They were merely heated for 1 hr at the temperatures shown in Table IV and then quenched. W1 steel was water quenched while the others were oil quenched. The microstructures and hardness data were used as criteria for determining the lower critical temperatures.

The term "spheroidizing temperature" will always mean a temperature about 25 to 50°F below the lower critical temperature and is a constant for each individual steel. The actual spheroidizing temperatures used for these steels are given in Table II.

TABLE III.

The Hardness of A2 Steel After Tempering 4 Hr at Various Temperatures

Tempering Temperature °F	Hardness RC
400	57
600	54
800	53
1000	50.5
1200	35.5
1400	29
1500	60

TABLE IV.

As-quenched Hardnesses for Various Tool Steels

Quenching Temperature, °F	Rockwell C Hardness			
	H13	D3	M2	W1
1300				24
1325				23
1350		38		61
1400		35		65
1450		34	34	
1475		34	35	
1500	31	53	42	
1550	32		45	
1600	46			

C. Spheroidization Treatment:

Attempts to enhance the rate of spheroidization involved five procedures. They are described in detail in the rest of this chapter. All the steels were studied essentially in the same way. Any modification made in the treatments for an individual steel was suggested by the results of spheroidization obtained by these five procedures.

The hardness and microstructure of a sample of each grade of steel annealed at the tool steel mill were used as standards to which the various samples were compared. The samples annealed by the mill will be referred to as "mill samples." The mill samples were not from the same heats as the steels studied in this investigation. The mill sample hardnesses and the hardness specifications of the manufacturer for annealed steels of the various grades studied are given in Table V.

It can be seen from Table V that mill sample hardnesses of H13 and W1 steels fall below the specified range. Apparently, the mill tries to achieve the lowest possible hardness.

1. Cold Work Prior to Spheroidization: Bars about 10" long of each steel, except W1, were softened by heating at their spheroidizing temperatures for 2 hr and furnace cooling. Each bar was then swaged down to 0.350" diameter in three steps; 0.450", 0.397", and 0.350". The effective reduction in area at these sizes were 19%, 37%, and 51% respectively. Five pieces, each about ½" in length, were cut from each of

TABLE V.

As-annealed Hardness Specifications and Mill Sample Hardnesses

Steel	Mill Sample Hardness	As-annealed hardness specifications
A2	15 RC	16-21 RC
H13	4 RC	12-18 RC
D3	22 RC	20-25 RC
M2	18 RC	15-21 RC
W1	81 RB	82-91 RB

these different diameter rods. Samples were also cut from the softened rods before they were swaged.

These cold worked samples of different sizes as well as softened but unworked samples of each steels were heated at their respective spheroidizing temperatures for 1 hr, 2 hr, 4 hr, and 8 hr followed by air cooling. The effect of rate of cooling was determined by heating another set of samples at the spheroidizing temperature for 8 hr and furnace cooling.

It was observed that the higher the deformation, and the longer the time at temperature, the better was the spheroidization. All the hardness data for these samples are given in Table VI. The first column in Table VI gives sample numbers which are arbitrary numbers identifying a set of samples. Thus, A2-1 represents a set of samples of A2 steel in which an individual sample will be identified by its size, e.g., A2-1(0.350" dia). The last four columns in Table VI give the hardnesses of the four samples in each set.

Hardnesses of the best swaged and annealed samples were a few points harder than those of the mill samples, and while they were spheroidized their carbides were somewhat smaller than those in the mill samples, Figs. 2 to 5.

a. <u>Cold Working and Spheroidizing from Different Original Microstructures</u>: The effect of original microstructure prior to cold working was studied by developing various microstructures in W1 steel before cold working. The

TABLE VI

Hardness of Samples Cold Worked Prior to Annealing

Sample No.	Ts, ^{o}F	Ts, hr	Cooling Medium	Rockwell C Hardness 0.350"	0.397"	0.450"	0.500"
A2-1	1425	1	air	26	26	27	30
A2-2	1425	2	air	26	25	26	29
A2-3	1425	4	air	23	24	25	27
A2-4	1425	8	air	22	24	25	26
A2-5	1425	8	furnace	18	20	20	25
H13-1	1525	1	air	18	19	21	23
H13-2	1525	2	air	16.5	17	20	22
H13-3	1525	4	air	13	13.5	16.5	19
H13-4	1525	8	air	10	10	11.5	17
H13-5	1525	8	furnace	6	7.5	8	16
D3-1	1460	1	air	27.5	29.5	30	32
D3-2	1460	2	air	26.5	27.5	28.5	30
D3-3	1460	4	air	26.5	27	28	28.5
D3-4	1460	8	air	25.5	26	27	27.5
D3-5	1460	8	furnace	23	24	26.5	27
M2-1	1475	1	air	28	28.5	29	32
M2-2	1475	2	air	26	27	28	30
M2-3	1475	4	air	25.5	25.5	27	29
M2-4	1475	8	air	25.5	25	26.5	27.5
M2-5	1475	8	furnace	23	24	26	27

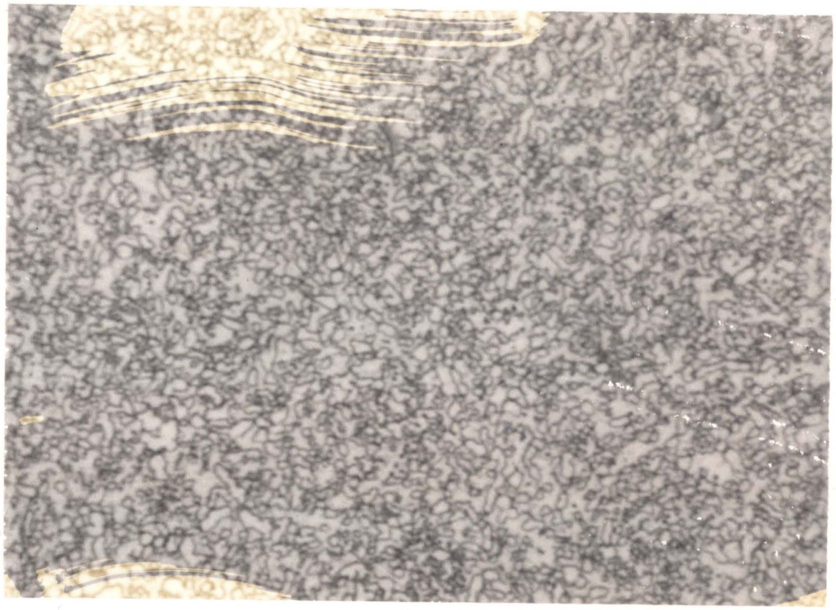

Fig. 2a: Mill Sample of A2 Steel.
Sample No. A2-00. A2 steel as annealed in a tool steel mill. 15 RC.

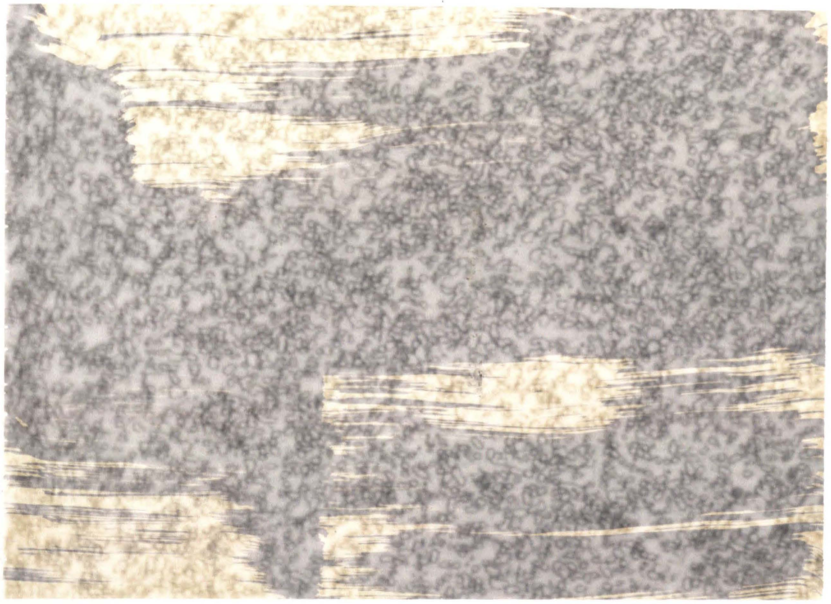

Fig. 2b: Cold Worked and Annealed Sample of A2 Steel.
Sample No. A2-5(0.350" dia.). Sample swaged to 0.350" dia. annealed 8 hr at 1425°F and furnace cooled. 18 RC.

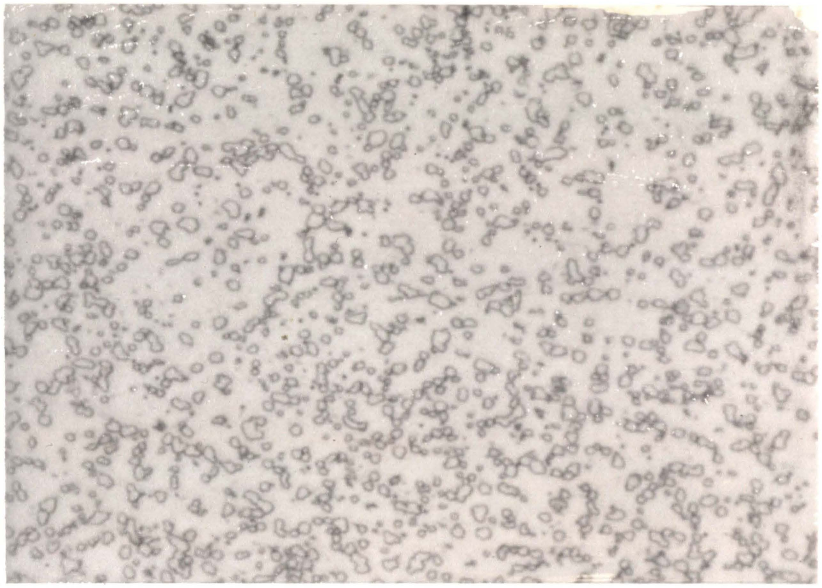

Fig. 3a: Mill Sample of H13 Steel.
Sample No. H13-00. H13 steel as annealed in a tool steel mill. 4 RC.

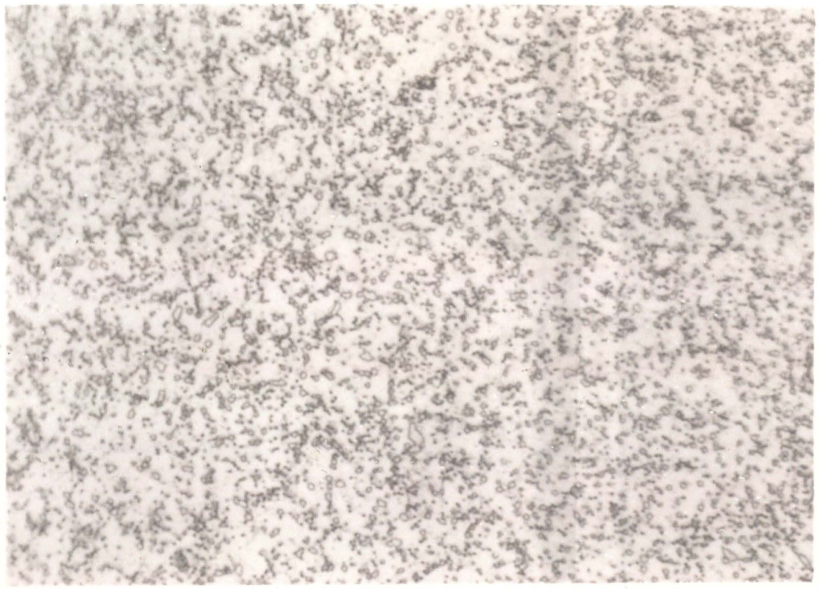

Fig. 3b: Cold Worked and Annealed Sample of H13 Steel.
Sample No. H13-5(0.350" dia.). Sample swaged to 0.350" dia. annealed 8 hr at 1525°F and furnace cooled. 6 RC.

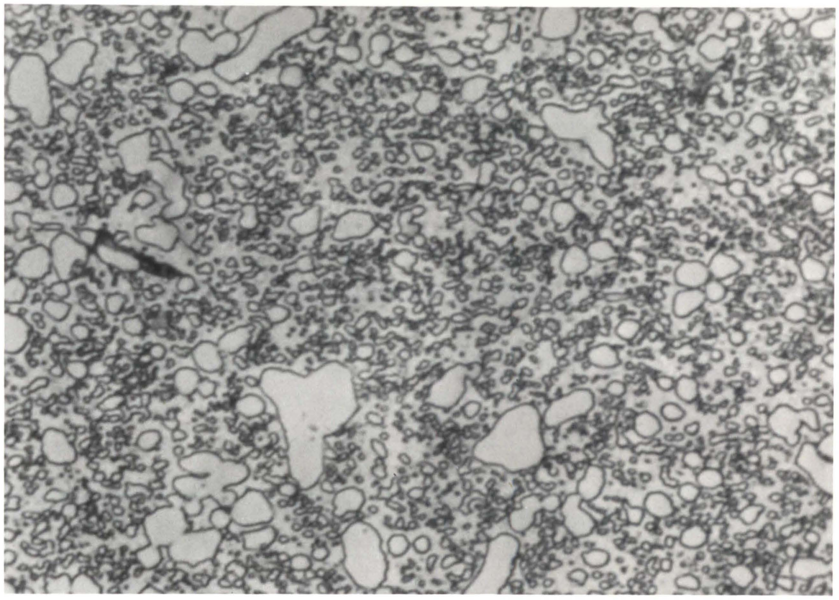

Fig. 4a: Mill Sample of D3 Steel. Sample No. D3-00. D3 steel as annealed in a tool steel mill. 22 RC.

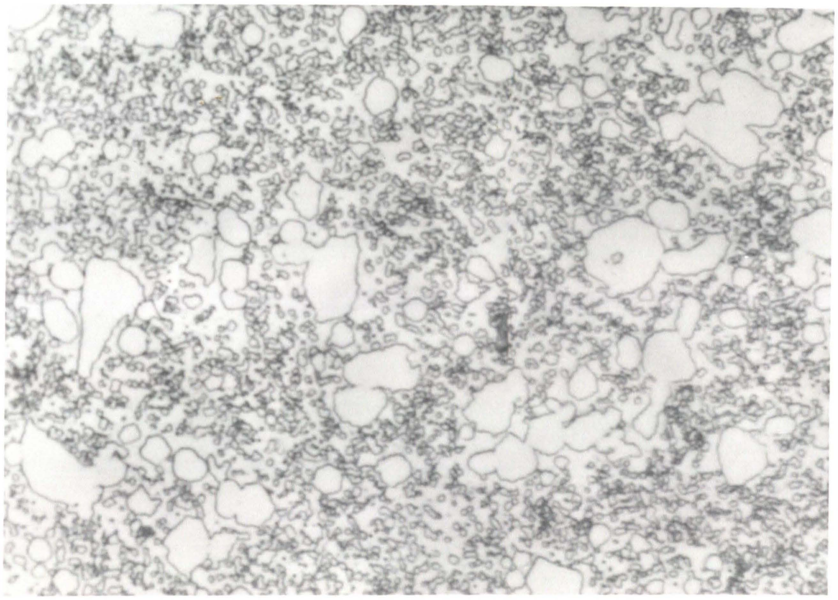

Fig. 4b: Cold Worked and Annealed Sample of D3 Steel. Sample No. D3-5(0.350" dia.). Sample swaged to 0.350" dia. annealed 8 hr at 1460°F and furnace cooled. 23 RC.

Fig. 5a: Mill Sample of M2 Steel.
Sample No. M2-00. M2 steel as annealed in a tool steel mill. 18 RC.

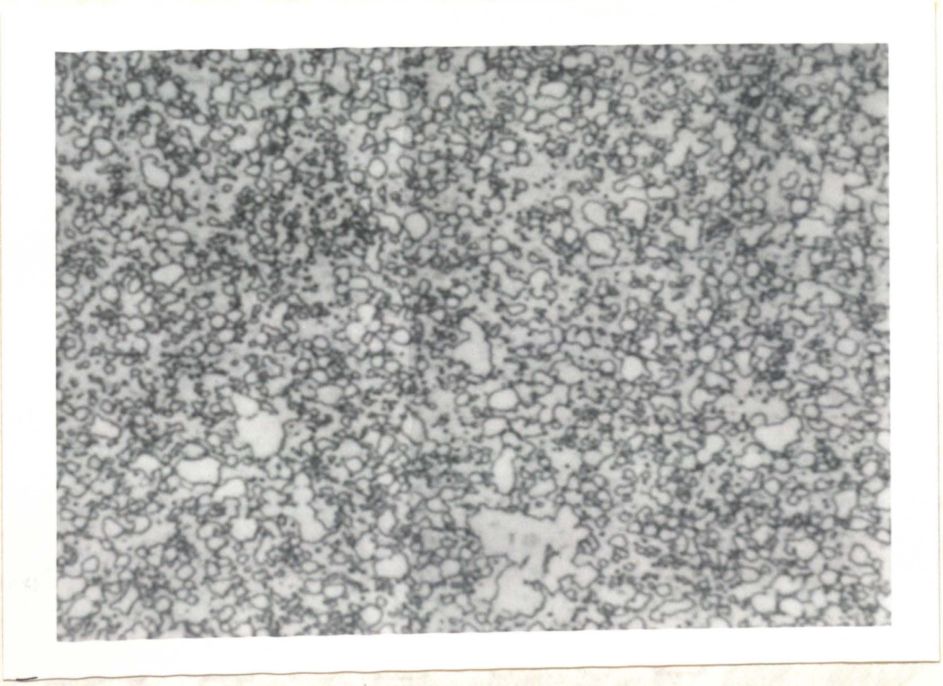

Fig. 5b: Cold Worked and Annealed Sample of M2 Steel.
Sample No. M2-5(0.350" dia.). Sample swaged to 0.350" dia. annealed 8 hr at 1475°F and furnace cooled. 23 RC.

as-received structure of W1 steel was a fine pearlite. The results obtained with this structure were compared with those obtained with two other structures, coarse pearlite and partially spheroidized pearlite. The coarse pearlite was obtained by heating a piece of W1 steel at 1800°F for 3 hr and furnace cooling. This piece developed a hardness of 94 RB. The partially spheroidized microstructure was obtained by heating another piece of W1 steel at 1325°F for 12 hr and air cooling. The hardness of this material was 93 RB. Both these pieces were swaged as before to get two sets of samples of each diameter, 0.450", 0.397", and 0.350" dia. One set of samples was spheroidized for 4 hr and air cooled; the other set was annealed 8 hr and was furnace cooled. Fig. 6 gives the microstructures of the spheroidized W1 steel samples which had different original microstructures prior to cold work. The related hardness data are given in Table VII.

2. Cycling Method: A pair of samples of all the steels, except W1, was heated independently at 1800°F for 1 hr and then quickly transferred to a furnace at the spheroidizing temperatures and held there for 2 hr. One sample from each pair was allowed to air cool while the other was reheated to 1800°F for another hour and then given another 2 hr at its spheroidizing temperature before air cooling. All the samples developed martensitic structures with various amounts of undissolved carbides. This was expected because the S-curves for all these steels, Fig. 7, indicate

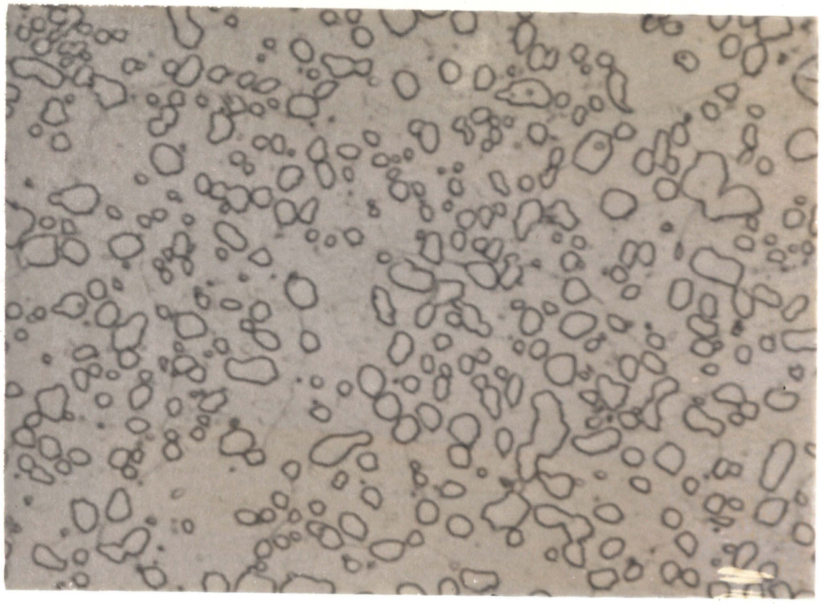

Fig. 6a: Mill Sample of W1 Steel.
Sample No. W1-00. W1 as annealed in a tool steel mill. 81 RB.

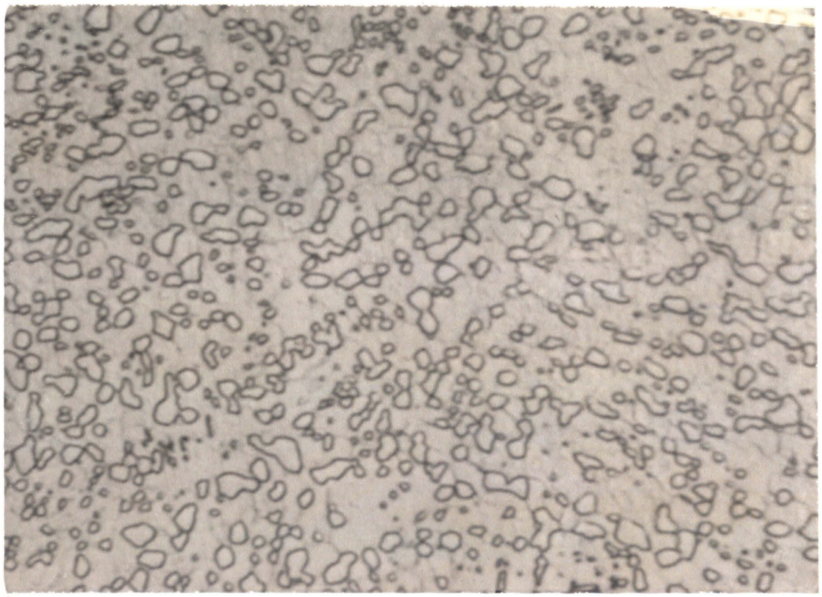

Fig. 6b: W1 Steel Annealed After Cold Working an As-received Bar.
Sample No. W1-5(0.350" dia.). Sample swaged to 0.350" dia. annealed 8 hr at 1325°F and furnace cooled. 90 RB.

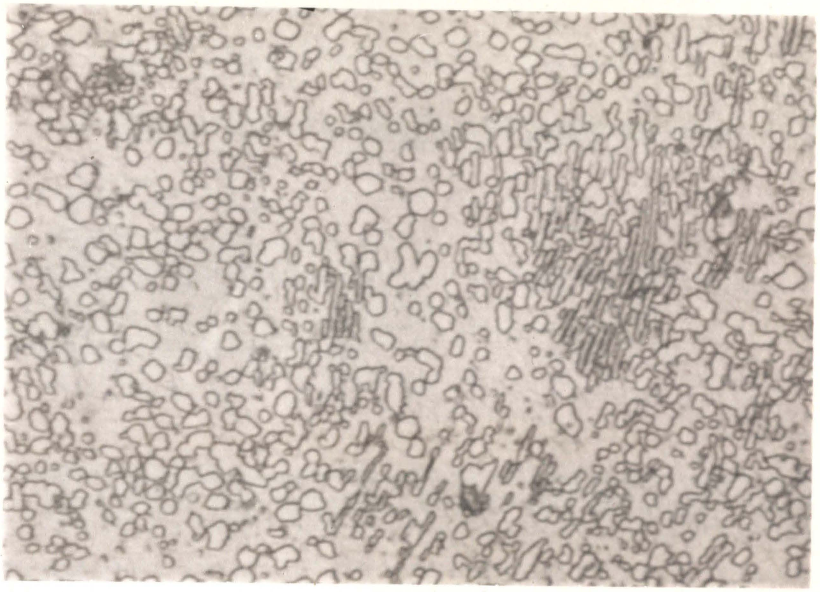

Fig. 6c: W1 Steel Annealed After Cold Working a Sample Containing Coarse Pearlite.
Sample No. W1-7(0.350" dia.). 87.5 RB

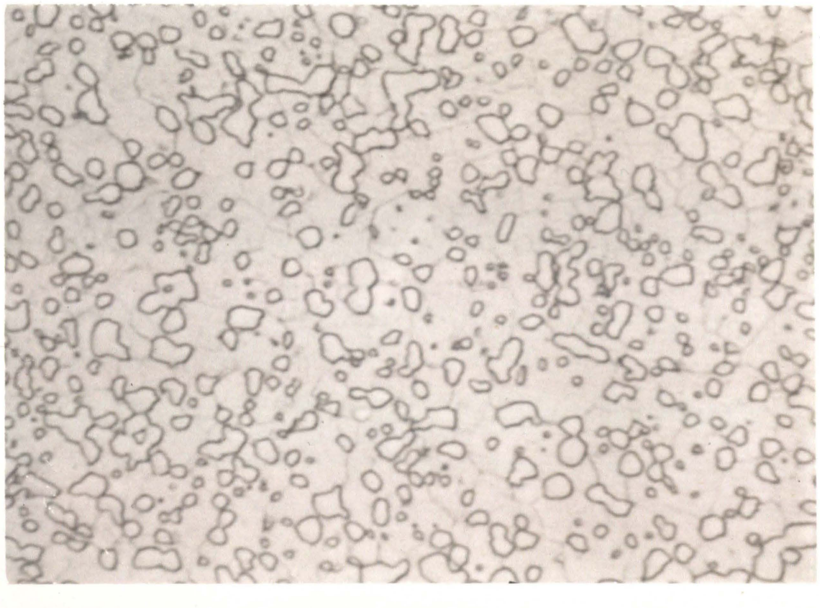

Fig. 6d: W1 Steel Annealed After Cold Working a Sample Containing Partially Spheroidized Pearlite.
Sample No. W1-9(0.350" dia.). 85 RB.

TABLE VII.

The Effect of Microstructure Prior to Cold Work on the Hardness of Annealed W1 Steel

Sample No.	Microstructure prior to cold work	t_s, hr	Cooling medium	Hardness, Rockwell B			
				0.350"	0.397"	0.450"	0.500"
W1-1	Fine pearlite	1	air	97	97.5	98.5	99
W1-2	Fine pearlite	2	air	96	96	97.5	98
W1-3	Fine pearlite	4	air	94	95.5	96	96.5
W1-4	Fine pearlite	8	air	92	92.5	92.5	94
W1-5	Fine pearlite	8	furnace	90	91.5	92	93
W1-6	Coarse pearlite	4	air	89	89.5	91	92
W1-7	Coarse pearlite	8	furnace	87.5	88.5	90	90.5
W1-8	P.S.P*	4	air	87	88	90	91
W1-9	P.S.P*	8	furnace	85	86	87.5	89

*Refer to the nomenclature on p. x

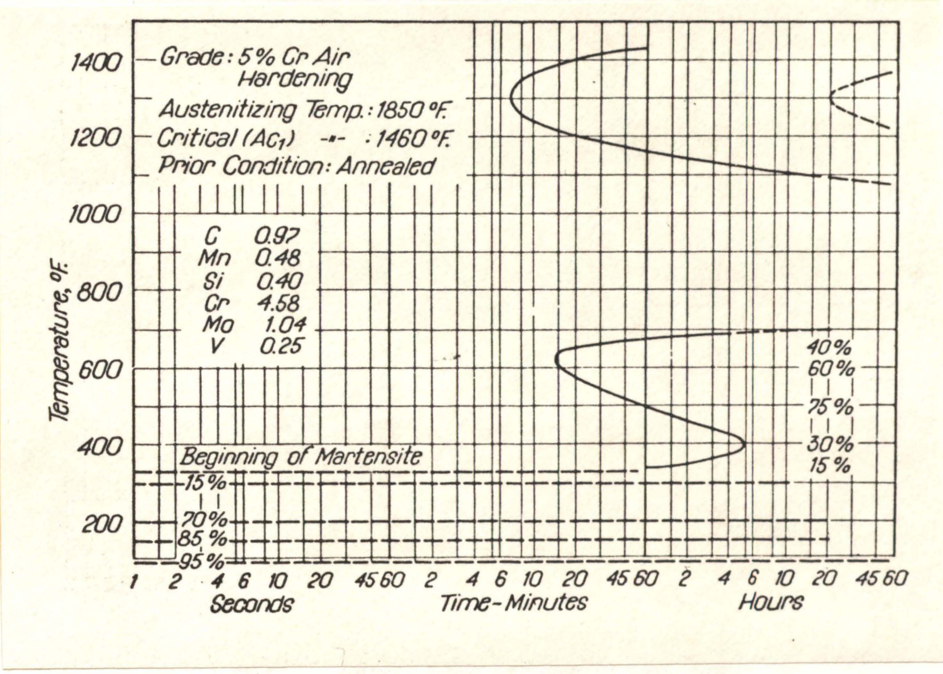

Fig. 7a: S-Curve for A2 Steel.

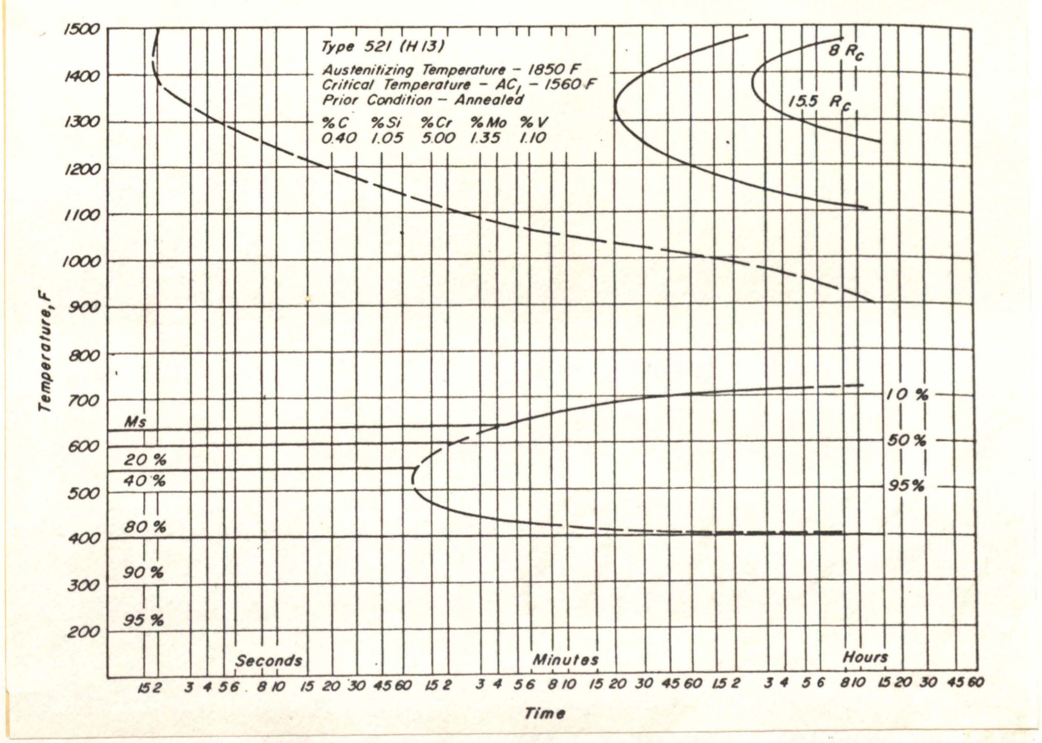

Fig. 7b: S-Curve for H13 Steel.

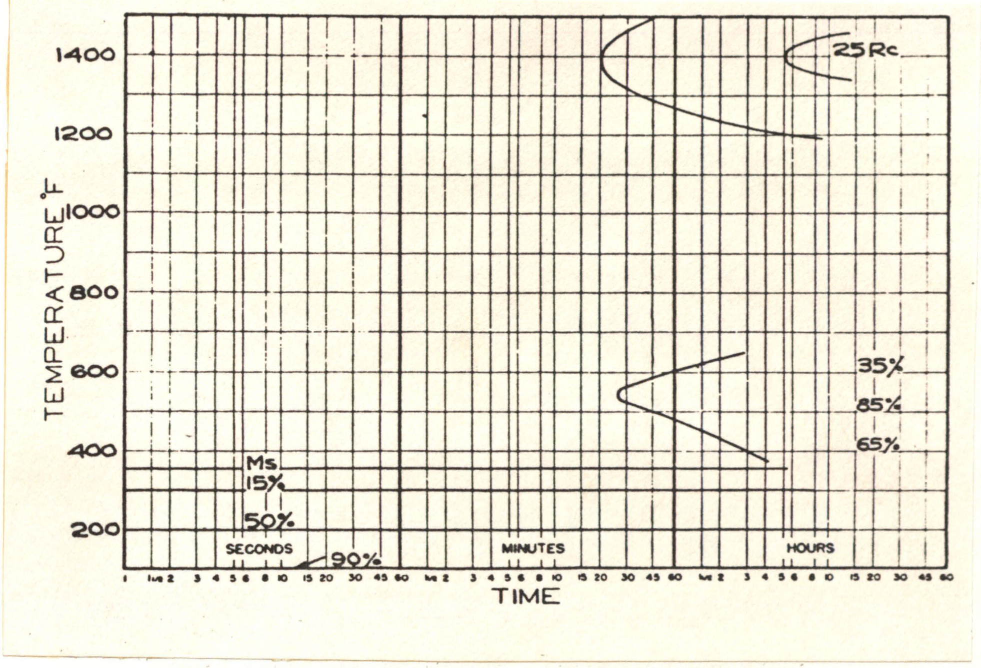

Fig. 7c: S-Curve for M2 Steel.

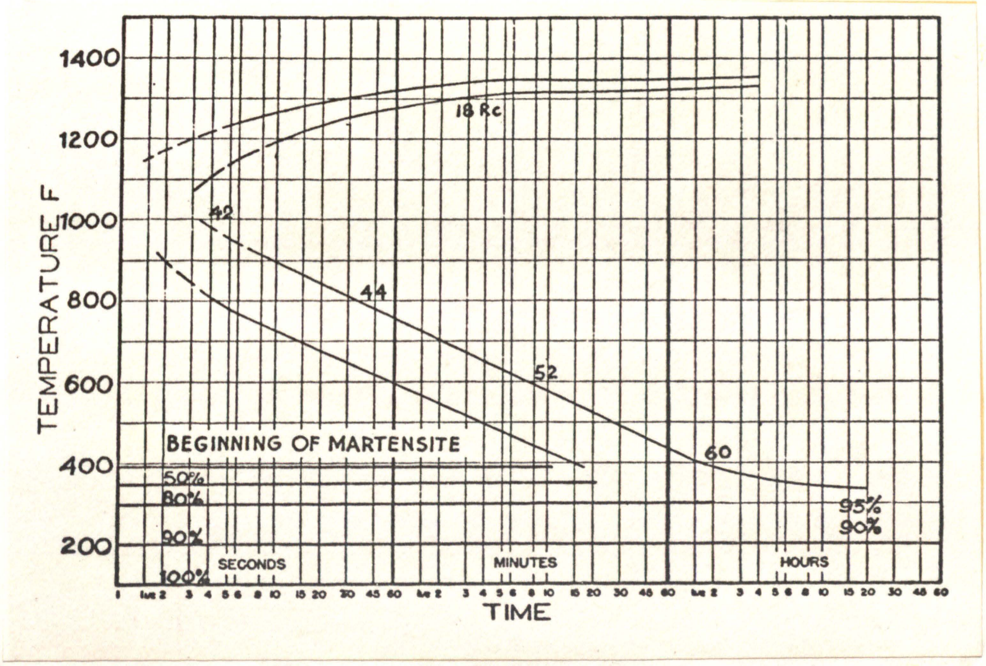

Fig. 7d: S-Curve for W1 Steel.

that 2 hr at the spheroidizing temperatures, which were very close to the lower critical temperatures, was too short a time for complete transformation of the austenite, and all these steels have sufficient hardenability to air harden.

The samples were then tempered at their spheroidizing temperatures for 6 hr and air cooled. After this treatment, all the samples showed a spheroidized microstructure. It was noticed that the two cycle samples, heated to 1800°F twice, of all the steels showed coarser carbides than the single cycle samples, Fig. 8. Their hardnesses, given in Table VIII, confirmed this.

a. Cycling Method for W1 Steel: The carbon tool steel, type W1, was not austenitized at 1800°F along with the alloy tool steels because that high an austenitizing temperature would cause it to transform to a coarse pearlite rather than a spheroidal carbide dispersion. A pair of W1 samples was, therefore, austenitized at 1450°F for 1 hr and then quickly transferred to a furnace at its spheroidizing temperature, 1325°F, and held there for 2 hr. One of the samples was then allowed to cool in air while the other was reheated at 1450°F for another hour and then given another 2 hr at 1325°F before air cooling. Both the samples showed finely spheroidized microstructures with hardnesses of 86 RB and 83 RB, respectively. These samples were then heated again to 1325°F for 4 hr and air cooled. The two cycle sample, heated to 1450°F twice, showed much coarser carbides than the single cycle

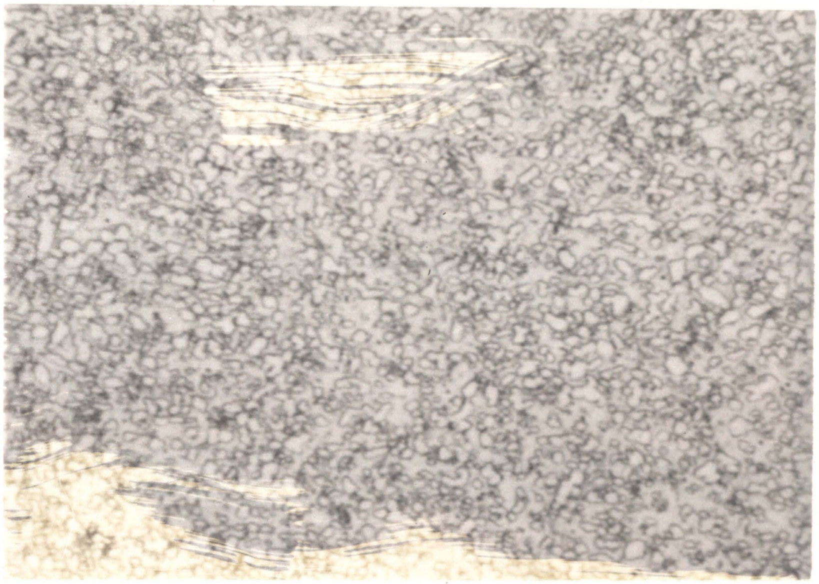

Fig. 8a: Two-Cycle Sample of A2 Steel.
Sample No. A2-22. Sample held 1 hr at 1800°F, 2 hr at 1425°F, 1 hr at 1800°F, 2 hr at 1425°F, air cooled and then reheated 6 hr at 1425°F. 17 RC.

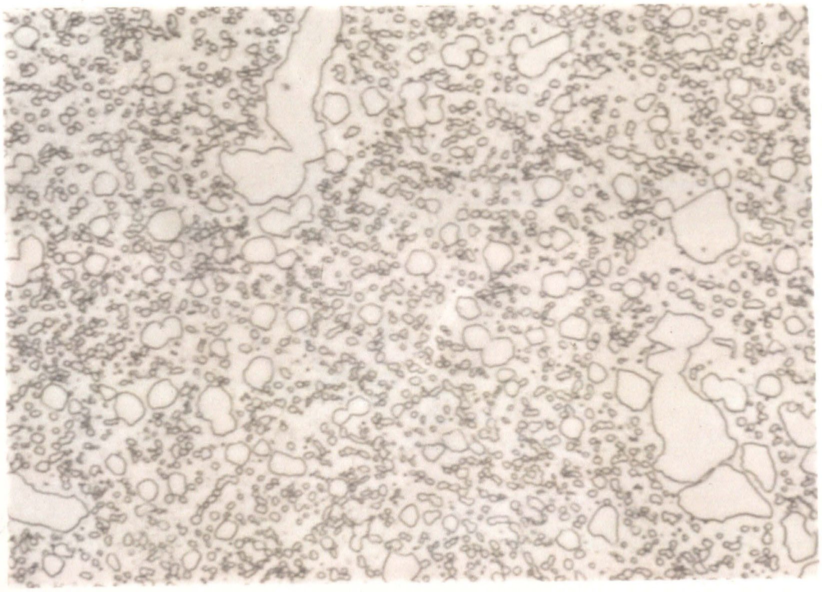

Fig. 8b: Two-Cycle Sample of D3 Steel.
Sample No. D3-22. Sample held 1 hr at 1800°F, 2 hr at 1460°F, 1 hr at 1800°F, 2 hr at 1460°F, air cooled and then reheated 6 hr at 1460°F. 21 RC.

Fig. 8c: One-Cycle Sample of H13 Steel. Sample No. H13-21. Sample held 1 hr at 1800°F, 2 hr at 1525°F, air cooled and then reheated 6 hr at 1525°F. 8.5 RC.

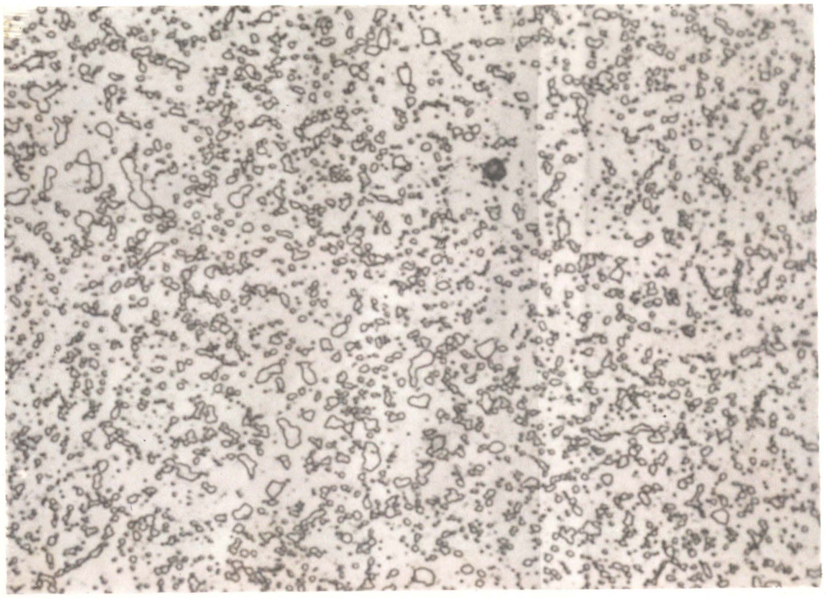

Fig. 8d: Two-Cycle Sample of H13 Steel. Sample No. H13-22. Sample held 1 hr at 1800°F, 2 hr at 1525°F, 1 hr at 1800°F, 2 hr at 1525°F, air cooled and then reheated 6 hr at 1525°F. 7.5 RC.

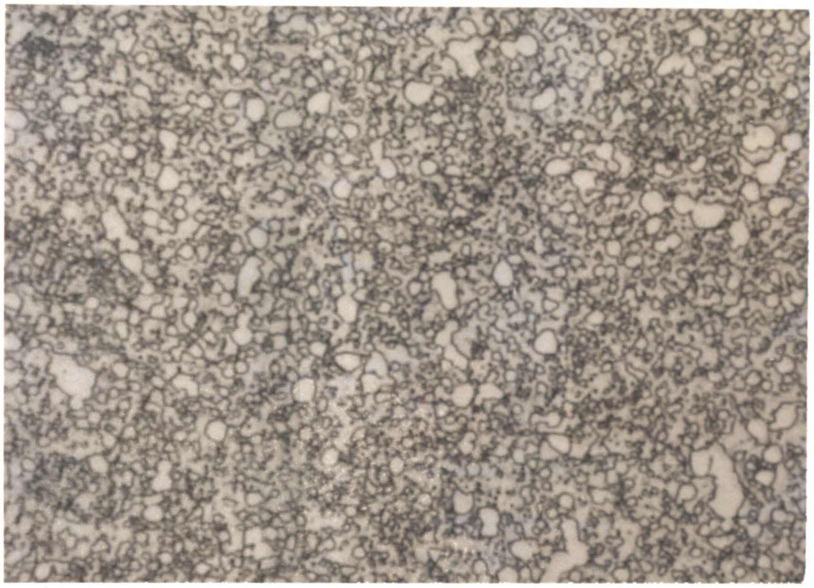

Fig. 8e: Two-Cycle Sample of M2 Steel.
Sample No. M2-22. Sample held 1 hr at 1800°F, 2 hr at 1475°F, 1 hr at 1800°F, 2 hr at 1475°F, air cooled and then reheated 6 hr at 1475°F. 23 RC.

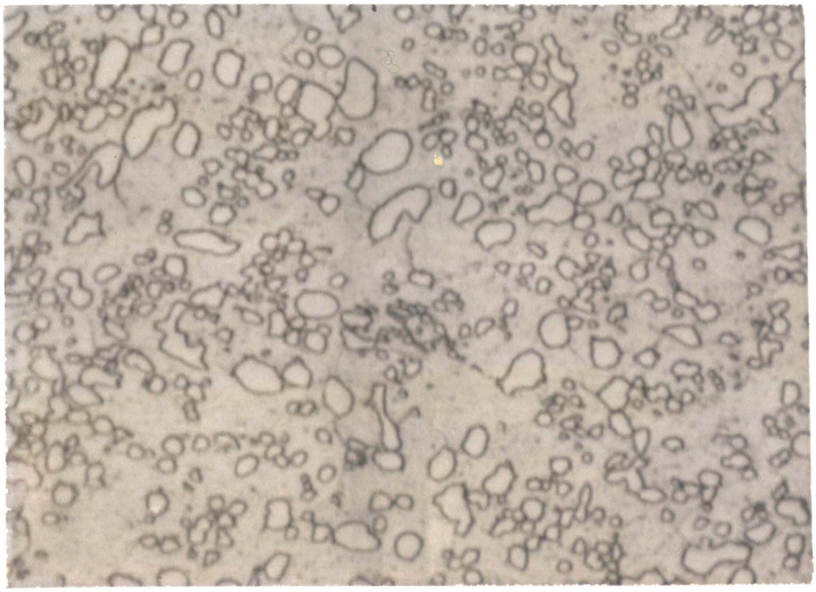

Fig. 8f: Two-Cycle Sample of W1 Steel.
Sample No. W1-22. Sample held 1 hr at 1450°F, 2 hr at 1325°F, 1 hr at 1450°F, 2 hr at 1325°F, air cooled and then reheated 6 hr at 1325°F. 82 RB.

TABLE VIII.

Hardness Data for Cycling Method

Single Cycle		Double Cycle	
Sample No.	Hardness	Sample No.	Hardness
A2-21	23 RC	A2-22	17 RC
H13-21	8.5 RC	H13-22	7.5 RC
D3-21	22 RC	D3-22	21 RC
M2-21	26 RC	M2-22	23 RC
W1-21	84 RB	W1-22	82 RB

sample, Fig. 8f. The final hardnesses were 82 RB and 84 RB, respectively as shown in Table VIII.

b. Modification of Cycling Method for H13 Steel: It can be seen that there is a range of carbide sizes in sample No. H13-21, Fig. 8c. This suggested a modification in the cycling method. If such a material were held for the proper time at a suitable austenitizing temperature, only the smaller carbides might be dissolved and the larger ones would not be completely dissolved. On subsequent transformation at the spheroidizing temperature, the undissolved carbide nucleii might serve as sites for carbide precipitation and with the same or less time as that given to sample H13-22 at the spheroidizing temperature, it might be possible to get coarser spheroids. The actual heat treatment was as follows:

Three as-received samples were heated to 1800°F for 1 hr and then transferred to a furnace at 1525°F. These three samples, H13-23, H13-24, and H13-25, were held at 1525°F for 2 hr, 4 hr, and 8 hr, respectively, and then air cooled. After checking their hardness and microstructure they were heated again to 1525°F for 6 hr and air cooled. Note that H13-21 and H13-23 were given identical treatments.

Eight more samples were treated in exactly the same way as H13-21. They were divided into two sets of four samples each. The samples in the first set were heated to 1800°F for 5 min, 15 min, 30 min and 1 hr and then transferred to a furnace at 1525°F where they were held for 2 hr. The second set samples were treated in the same way at 1800°F but held for 8 hr at 1525°F. All the samples were then air cooled. All the samples were heated again to 1525°F for 8 hr and air cooled. The hardness data for all these samples are given in Table IX and Table X.

Figs. 9a and 9b give microstructures of the representative samples of the two sets. The hardness data and the microstructures of these samples indicated that the spheroidization in these samples was not much better than in sample No. H13-21, Fig. 8c, and sample No. H13-22, Fig. 8d. Sample No. H13-33 did show some massive carbide particles which were even larger than those in the mill sample. But, the carbide particles in the mill sample were uniform in size whereas those in sample No. H13-33 and the other 8 hr samples were nonuniform.

Sample No. H13-26, Fig. 9a, was typical of the set of 2 hr samples and it did not contain any exceptionally massive carbide particles. In both 2 hr and 8 hr samples it was observed that the variation in the austenitizing time did not seem to cause any appreciable change in the hardness or the microstructure.

3. Isothermal Annealing: A pair of samples of each steel, except W1, was austenitized at 1650°F for 1 hr and then quickly transferred to another furnace at the spheroidizing temperature. They were held at the spheroidizing temperature for 24 hr and then one sample from each pair was air cooled while the other was furnace cooled. The S-curves of

TABLE IX.

Hardness Data for the Modified Cycling Method for H13 Steel

Sample No.	Time at 1525°F before tempering, hr	Hardness after tempering, RC
H13-23	2	8.5
H13-24	4	8
H13-25	6	8

TABLE X.

Additional Hardness Data for the Modified Cycling Method for H13 Steel

Sample No.	Time at 1800°F, min.	Time at 1525°F before tempering, hr	Hardness after tempering, RC
H13-26	5	2	14
H13-27	15	2	16
H13-28	30	2	15.5
H13-29	60	2	15
H13-30	5	8	10
H13-31	15	8	12
H13-32	30	8	11
H13-33	60	8	9

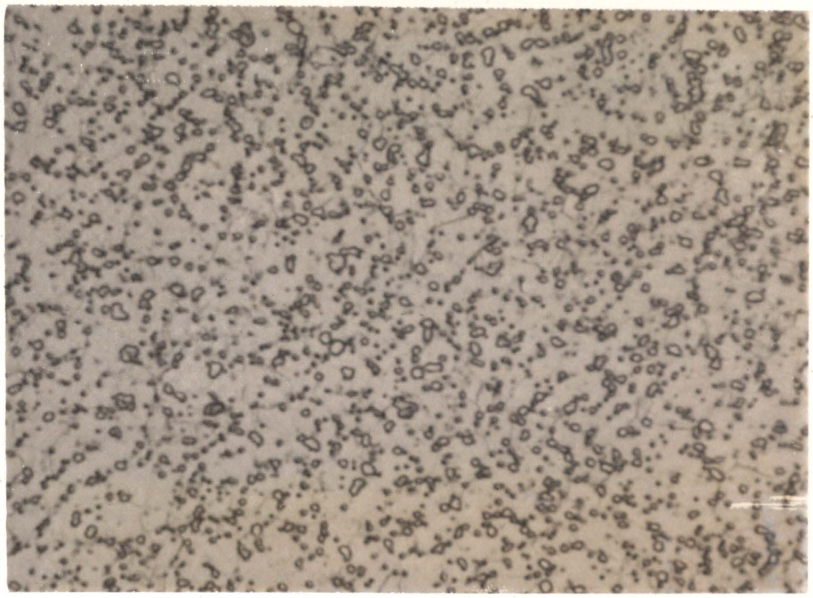

Fig. 9a: Sample H13-26 Treated by Modified Cycling Method. 14 RC.

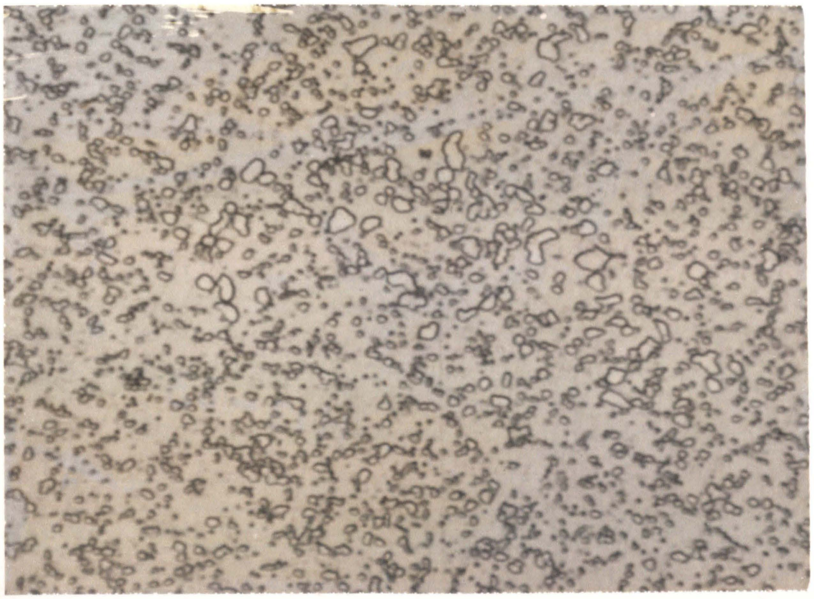

Fig. 9b: Sample H13-33 Treated by Modified Cycling Method. 9 RC.

A2 and M2 steels, Figs. 7a and 7c, show that 24 hour at their spheroidizing temperatures are insufficient for complete transformation of austenite. As a result, the air cooled samples of A2 and M2 are somewhat hard, 51 RC and 32 RC, respectively, and showed martensite. The furnace cooled samples of these steels had carbide spheroids and a dark-etching transformation product that was apparently rather soft. The air cooled samples of H13 and D3 steels were soft, 6 RC and 22 RC, respectively, and showed finely spheroidized microstructures. The furnace cooled sample of D3 showed only traces of a dark-etching transformation product whereas the H13 steel sample was fully spheroidized and did not show any dark-etching microconstituent.

Another pair of samples of each steel was isothermally annealed for 32 hr at their spheroidizing temperatures. As before, one sample from each pair was air cooled and the other furnace cooled. With more time, more austenite transformed and hence there was less martensite in the air cooled samples and less dark-etching transformation product in the furnace cooled samples. In fact, A2 and D3 showed only traces and H13 and M2 did not show any dark-etching product in the furnace cooled samples, Fig. 10.

At the nose of the S-curve, the transformation starts and finishes at the fastest rate. A pair of samples of each steel, except D3 whose nose temperature was not known, was, therefore, isothermally annealed at their respective upper nose temperatures for 32 hr. One sample from each pair was

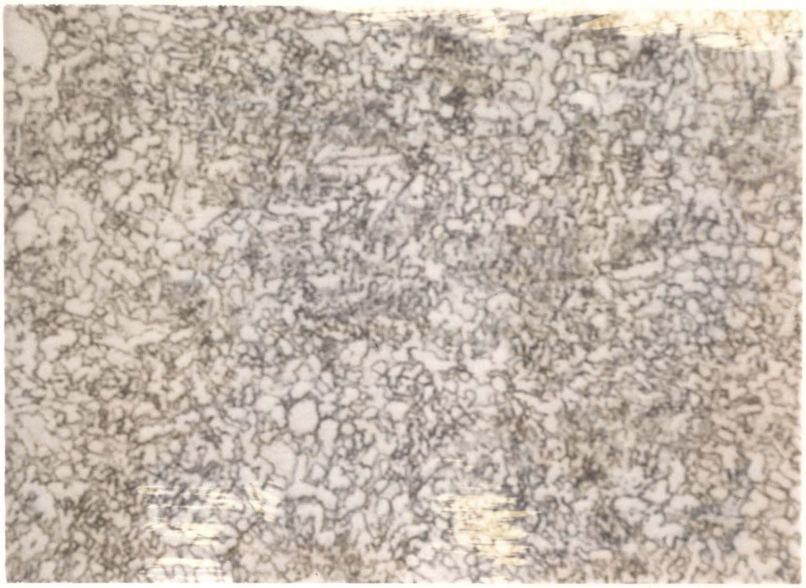

Fig. 10a: Isothermally Annealed Sample of A2 Steel. Sample No. A2-44. As-received sample austenitized at 1650°F for 1 hr, isothermally transformed at 1425°F for 32 hr and furnace cooled. 18 RC.

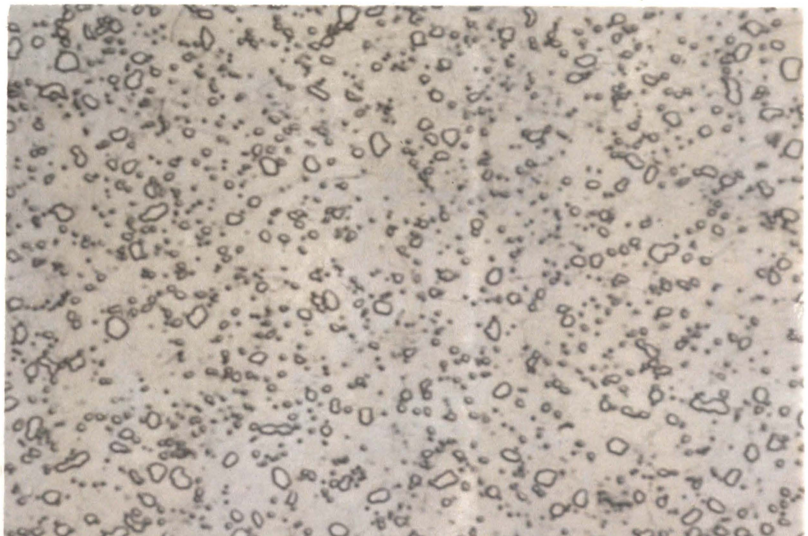

Fig. 10b: Isothermally Annealed Sample of H13 Steel. Sample No. H13-44. As-received sample austenitized at 1650°F for 1 hr, isothermally transformed at 1525°F for 32 hr and furnace cooled. 1 RC.

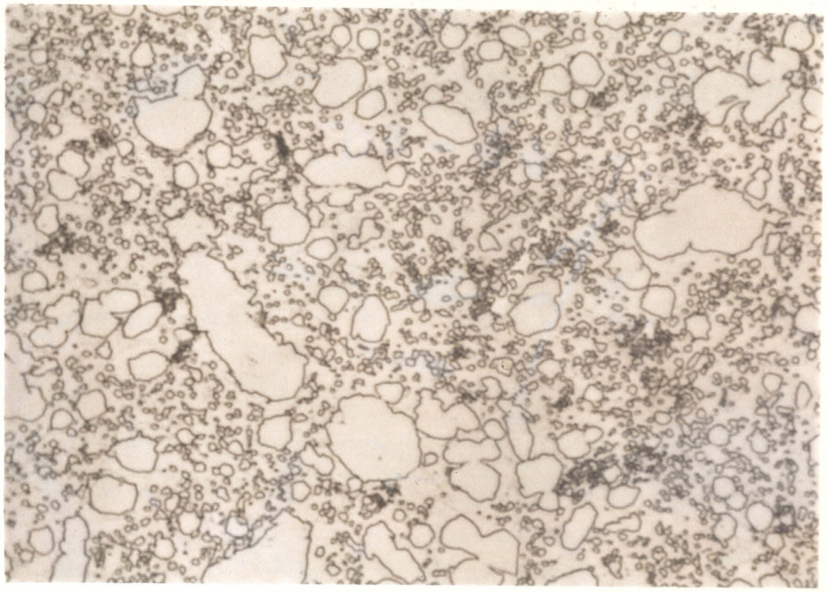

Fig. 10c: Isothermally Annealed Sample of D3 Steel. Sample No. D3-44. As-received sample austenitized at 1650°F for 1 hr, isothermally transformed at 1460°F for 32 hr and furnace cooled. 20.5 RC.

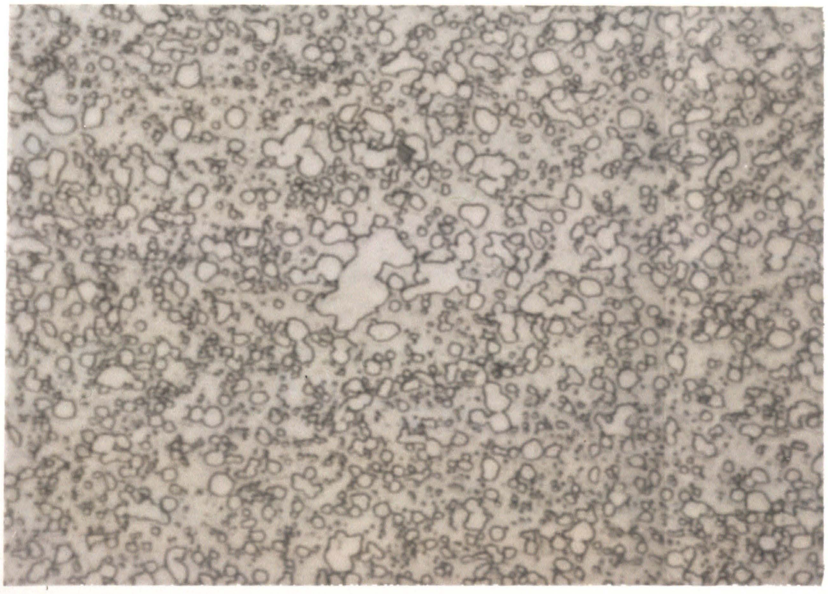

Fig. 10d: Isothermally Annealed Sample of M2 Steel. Sample No. M2-44. As-received sample austenitized at 1650°F for 1 hr, isothermally transformed at 1475°F for 32 hr and furnace cooled. 17 RC.

air cooled and the other was furnace cooled. The air cooled samples did not show any martensite. The furnace cooled samples showed a dark-etching product and carbide spheroids which were much finer than those in the samples isothermally annealed at their spheroidizing temperatures. This is an obvious effect of lowering the temperature of isothermal transformation. All the hardness data for the isothermal transformation samples are given in Table XI.

A slow rate of cooling in the transformation range gives more time for the sluggish transformation of austenite. This fact coupled with the fact that transformation is most rapid at the nose temperatures was made use of in the following modification of isothermal annealing.

a. Modified Isothermal Annealing: A sample of each steel, except D3 whose nose temperature was not known, was austenetized independently at 1650°F, 1400°F for W1 steel, for 1 hr, and then the samples were cooled down to the spheroidizing temperature in the furnace with power off. They were held at their spheroidizing temperatures for the times given in Table XII. The furnace was then cooled down to the proper upper nose temperature and held there for the times given in Table XII. The samples were then air cooled. All the samples showed some fine spheroids with some dark-etching microconstituent in the background. The hardness data of these samples are given in Table XII.

TABLE XI

Hardness Data for Isothermal Annealing

Sample No.	Transformation temperature, °F	Transformation time, hr	Cooling medium	Hardness RC
A2-41	1425	24	air	51
A2-42	1425	24	furnace	19
A2-43	1425	32	air	46
A2-44	1425	32	furnace	18
A2-45	1300	32	air	21
A2-46	1300	32	furnace	20
H13-41	1525	24	air	6
H13-42	1525	24	furnace	3.5
H13-43	1525	32	air	3
H13-44	1525	32	furnace	1
H13-45	1400	32	air	3
H13-46	1400	32	furnace	3
D3-41	1460	24	air	22
D3-42	1460	24	furnace	21.5
D3-43	1460	32	air	21
D3-44	1460	32	furnace	21
M2-41	1475	24	air	32
M2-42	1475	24	furnace	19
M2-43	1475	32	air	18
M2-44	1475	32	furnace	17
M2-45	1400	32	air	18
M2-46	1400	32	furnace	18

TABLE XII.

Hardness Data for Modified Isothermal Annealing

Sample No.	T_a, °F	T_s, °F	t_s, hr	T_n, °F	t_n, hr	Hardness
A2-50	1650	1425	2	1300	4	20 RC
H13-50	1650	1525	6	1400	4	3.5 RC
M2-50	1650	1475	4	1400	6	20 RC
W1-50	1400	1325	1	1050	1/2	85 RB

b. Isothermal Annealing of W1 Steel: Although the nature of the isothermal annealing treatment given W1 was the same as that given the other steels, there were some variations and some modifications. Hence, this treatment is discussed separately.

Seven samples of W1 steel were austenitized at 1400°F for 1 hr and five of them were transferred to a furnace at 1325°F. The other two were transferred to a furnace at the upper nose temperature, 1050°F. At 1325°F, three samples were held for 3 hr, 6 hr, and 12 hr and then air cooled. The other two were held for 3 hr and 6 hr and furnace cooled. At 1050°F, both the samples were held for 2 hr and then one of them was air cooled while the other was furnace cooled. Both the samples transformed at 1050°F showed fine carbides with some small colonies of pearlite. The sample, W1-45, that was transformed for 12 hr at 1325°F showed the coarsest carbides and was 83.5 RB. All the hardness data for these steels are given in Table XIII.

c. Modification in Isothermal Annealing of W1 Steel: This modification was made to study the effect of austenitizing temperature and time on isothermal annealing of W1 steel. Three samples were austenitized at 1400°F for different times, 2 hr, 4 hr, and 6 hr, transformed at 1325°F for 12 hr, and then air cooled. The sample austenitized for 2 hr, sample No. W1-51, showed coarse spheroids, Fig. 11a, which were coarser than those in the sample austenitized for 1 hr, sample No. W1-45. The other two samples also showed coarse

TABLE XIII.

Variation in Hardness of W1 Steel with Transformation Temperature and Time

Sample No.	Transformation temperature, °F	Transformation time, hr	Cooling medium	Hardness RB
W1-41	1325	3	air	86
W1-42	1325	3	furnace	85
W1-43	1325	6	air	85
W1-44	1325	6	furnace	84
W1-45	1325	12	air	83.5
W1-46	1050	2	air	93.5
W1-47	1050	2	furnace	91.5

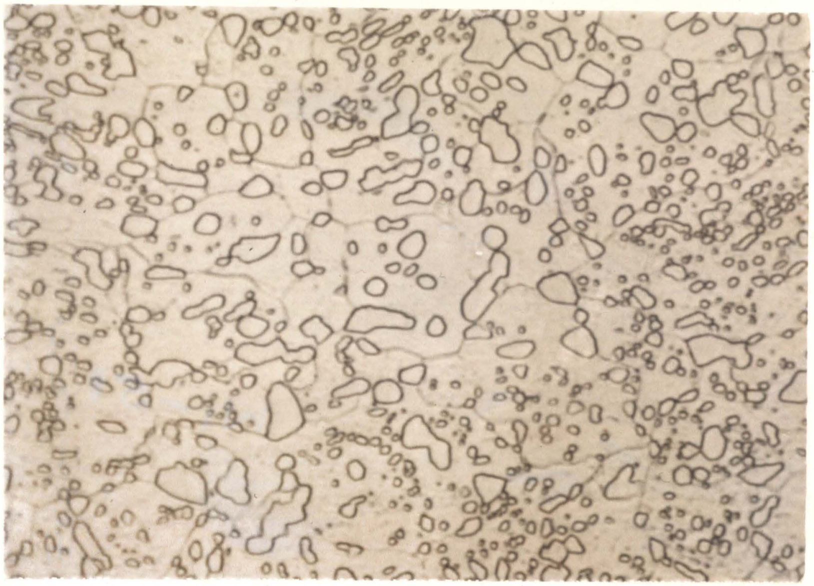

Fig. 11a: Sample No. W1-51.
Sample austenitized at 1400°F for 2 hr, isothermally transformed at 1325°F for 12 hr and air cooled. 82.5 RB.

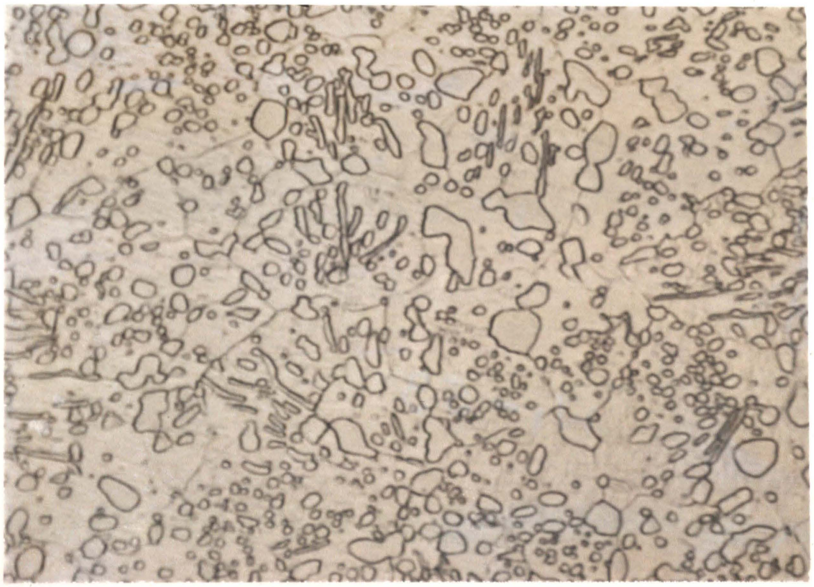

Fig. 11b: Sample No. W1-53.
Sample austenitized at 1400°F for 6 hr, isothermally transformed at 1325°F for 12 hr and air cooled. 83 RB.

spheroids but they showed a tendency to form very coarse pearlite. The thick carbide lamellae in sample No. W1-53, austenitized for 6 hr, at 1400°F are shown in Fig. 11b.

Two more sets of three samples each were austenitized at 1500°F and 1600°F for 20 min., 40 min., and 1 hr each and then transformed at 1325°F for 12 hr, as before. They were all air cooled. 1600°F was observed to be high enough to homogenize the austenite and hence the isothermally annealed product was pearlitic rather than spheroidal. The samples austenitized at 1500°F were spheroidized, more or less, to the same degree of coarseness and were comparable with the samples austenitized at 1400°F for 2 hr. All the hardness data of these steels are given in Table XIV.

4. Martensite Formation Prior to Spheroidization: This treatment was essentially a high temperature tempering treatment. The quenching temperatures and tempering times were varied to get the most coarsely spheroidized structure. Some modification was done on W1 steel and hence it is discussed separately.

Seven samples from each of the other steels were oil quenched after austenitizing for 1 hr. Seven different austenitizing temperatures were used: 1500°F, 1550°F, 1600°F, 1650°F, 1700°F, 1800°F, and 1900°F. The hardness values of these samples after quenching from these temperatures are given in Table XV.

TABLE XIV.

Effect of Austenitizing Temperature and Time on Hardness of Isothermally Annealed W1 Steel

Sample No.	T_a, °F	t_a, hr	Hardness, RB
W1-51	1400	2	82.5
W1-52	1400	4	83
W1-53	1400	6	83
W1-54	1500	1/3	83.5
W1-55	1500	2/3	83.5
W1-56	1500	1	84
W1-57	1600	1/3	89
W1-58	1600	2/3	90
W1-59	1600	1	90

TABLE XV.

As-quenched and As-tempered Hardnesses

Hardening temp, °F	As-quenched hardness, RC				As-tempered hardness, RC			
	A2	H13	D3	M2	A2	H13	D3	M2
1500	46	31	53	42	24	13	26	25
1550	62	32	56	45	26	11	29.5	27
1600	62	46	57	51	25	10	29	25
1650	62	49	63	45	26	13.5	30	26
1700	61	50	61	43	27	12	29	26
1800	57	52	60	49	19	12.5	27	26
1900	58	52	59	59	22	13	29	24

All the hardened samples were then tempered at their respective spheroidizing temperature for 4 hr, and furnace cooled. Their hardness values also are given in Table XV. These tempered samples showed tempered martensite along with some fine carbides. The softest samples as seen from the hardness data in Table XV showed the coarsest carbides among all other samples of those particular steels. The effect of tempering time and rate of cooling from the tempering temperature were studied on samples which were austenitized like the samples that showed the coarsest spheroids in this series.

Three pairs of samples of each steel were quenched from the temperatures that gave lowest hardness and coarsest spheroids after tempering. For example, 1800°F was used for A2 steel. All samples were then tempered at their respective spheroidizing temperatures for 2 hr, 4 hr, and 8 hr. One sample from each pair was air cooled while the other was furnace cooled. Their hardness values are given in Table XVI. The degree of coarseness of the spheroids seemed to increase with increasing tempering time. Thus, the coarsest spheroids and the lowest hardness were shown by the samples tempered for 8 hr and furnace cooled, Fig. 12. Except for M2, the spheroids in these samples were much finer than those in the respective mill samples.

a. <u>Martensite Formation Prior to Spheroidization in W1 Steel</u>:

The effect of quenching medium on the hardened and tempered samples of W1 steel was studied. Two sets of

TABLE XVI.

Effect of Tempering Time on Hardness

Tempering time, hr	Cooling medium	As-tempered hardness, RC A2	H13	D3	M2
2	air	31	17	31	25
2	furnace	21	13	28	24
4	air	28	16	28	23
4	furnace	19	11	27	22
8	air	27	15	26	21
8	furnace	17	10	25	18

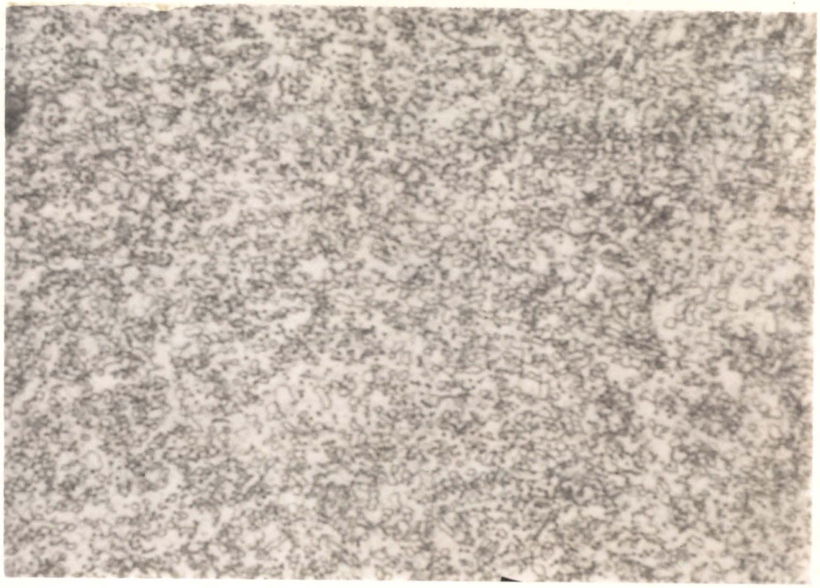

Fig. 12a: Hardened and Tempered Sample of A2 Steel. Sample No. A2-76. Sample heated at 1800°F for 1 hr and oil quenched. It was then tempered for 8 hr at 1425°F and furnace cooled. 17 RC.

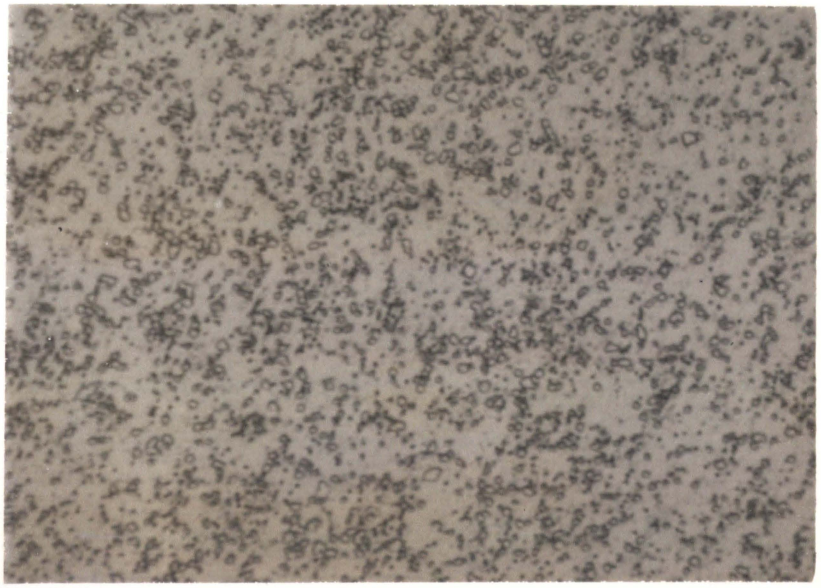

Fig. 12b: Hardened and Tempered Sample of H13 Steel. Sample No. H13-76. Sample heated at 1600°F for 1 hr and oil quenched. It was then tempered for 8 hr at 1525°F and furnace cooled. 9.5 RC.

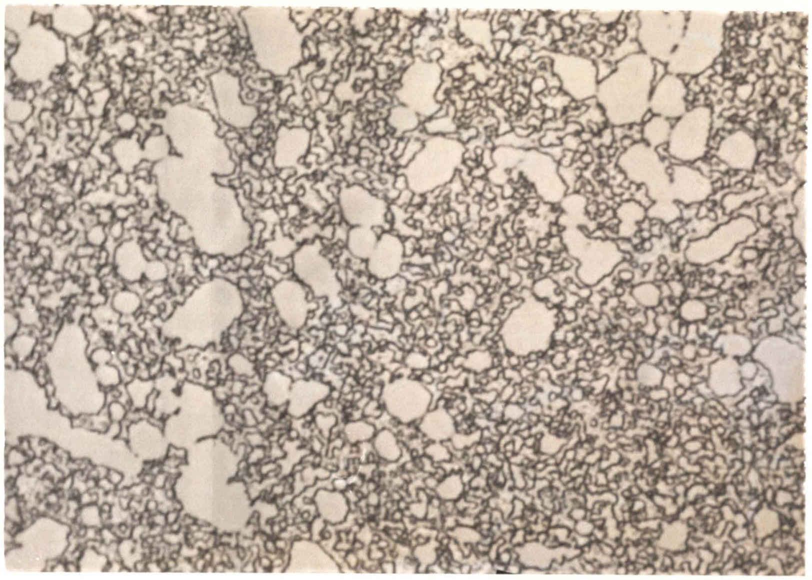

Fig. 12c: Hardened and Tempered Sample of D3 Steel. Sample No. D3-76. Sample heated at 1800°F for 1 hr and oil quenched. It was then tempered for 8 hr at 1460°F and furnace cooled. 25 RC.

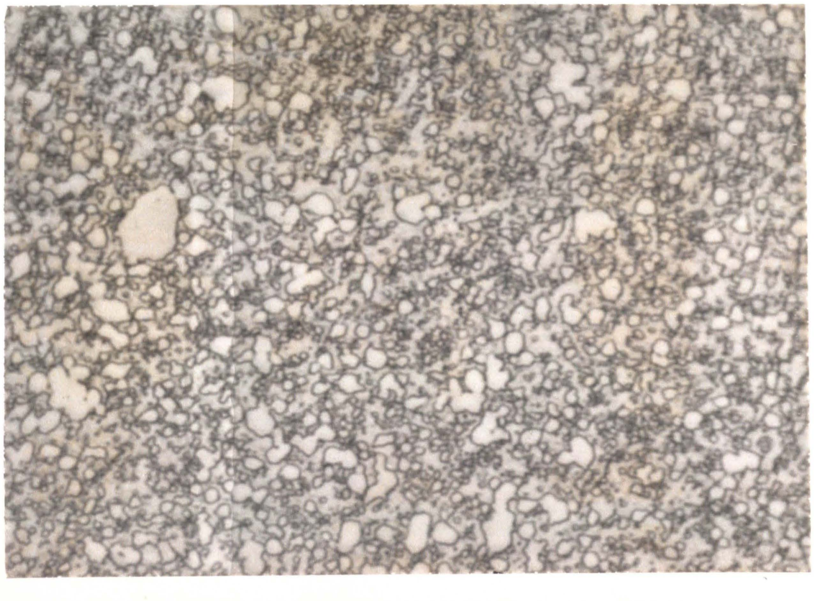

Fig. 12d: Hardened and Tempered Sample of M2 Steel. Sample No. M2-76. Sample heated at 1900°F for 1 hr and oil quenched. It was then tempered for 8 hr at 1475°F and furnace cooled. 18 RC.

five samples each, ½" diameter by ½" length, were austenitized at 1400°F, 1500°F, 1600°F, 1700°F, and 1800°F. One set of samples was then water quenched and the other oil quenched. All the samples developed martensitic structures. The water quenched samples were much harder than the oil quenched samples. Their hardness values are given in Table XVII.

All the hardened samples were subsequently tempered at 1325°F for 4 hr and furnace cooled. The oil quenched samples appeared to be better spheroidized and softer than the water quenched samples, Table XVII. In particular, the sample, oil quenched from 1500°F showed the coarsest spheroids after tempering.

The effect of tempering time and rate of cooling from the tempering temperature were studied with four samples that had been oil quenched from 1500°F and tempered at 1325°F. One sample was tempered for 2 hr, one for 4 hr, and two were tempered for 8 hr. One of the 8 hr samples was furnace cooled while the other three were air cooled. All the samples were spheroidized. The finest carbides and the maximum hardness were shown by the 2 hr, air cooled sample. The 8 hr, furnace cooled sample, Fig. 13, was the softest and showed the coarsest spheroids. The hardness data of all these samples are given in Table XVIII.

5. Radiation Damage Prior to Spheroidization: The effect of radiation damage was studied only with W1 steel because the alloying elements in the other steels form highly

TABLE XVII.

As-quenched and As-tempered Hardness of W1 Steel

Sample No.	Hardening temp, °F	Quenching medium	As-quenched hardness, RC	As-tempered hardness, RB
W1-61	1400	oil	36	89
W1-62	1500	oil	40	84
W1-63	1600	oil	46	90.5
W1-64	1700	oil	48	85
W1-65	1800	oil	48	93
W1-66	1400	water	65	91
W1-67	1500	water	64	90.5
W1-68	1600	water	64	93
W1-69	1700	water	63.5	92
W1-70	1800	water	63	91.5

TABLE XVIII.

Effect of Tempering Time on Hardness of W1 Steel

Sample No.	Tempering time, hr	Cooling medium	Hardness RB
W1-71	2	air	90
W1-72	4	air	86
W1-73	8	air	84
W1-74	8	furnace	83

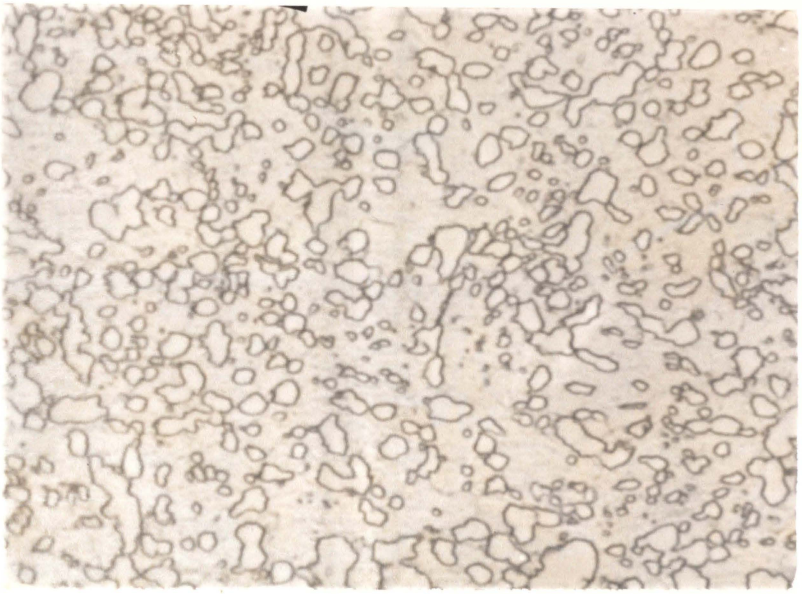

Fig. 13: Hardened and Tempered Sample of W1 Steel. Sample No. W1-74. Sample heated at 1500°F for 1 hr and oil quenched. It was then tempered for 8 hr at 1325°F and furnace cooled. 83 RB.

active isotopes with long half lives. The handling of highly active samples would have presented many problems. Furthermore, the results with W1 steel indicated that irradiation of tool steels prior to spheroidizing was not likely to be beneficial.

A rod of W1 steel was surface ground to remove the oxide scale. Nine pieces, about ½" in length, were cut from this rod. Their ends were ground off. All nine samples were then cleaned with methanol. They were divided into three sets of three samples each. Each set was placed in a plastic bag which in turn was placed in a cylindrical aluminum container. These cylinders were then sealed with epoxy resin.

The sealed containers were then irradiated in a swimming pool type reactor with a flux of 10^{12} neutrons/cm^2 for three different times: 3 hr, 6 hr, and 9 hr. The activity of these samples was then allowed to decay outside the reactor until the activity of each set of samples came down to about 20 mR. This decaying of activity to about 20 mR took about 9 weeks.

Microstructures of the samples irradiated for 3 hr and 9 hr were examined after polishing and etching with 5% nital. Their microstructures were fine pearlite and they appeared to be essentially the same as as-received samples of W1 steels. A polished sample of as-received W1 steel was then sealed in a Vycor tube along with a polished sample that had been irradiated for 3 hr. The tube had been evacuated before sealing. The tube was then heated at the spheroidizing temperature, 1325°F, for 1 hr. The sample irradiated for 9 hr was heated at 1325°F in exactly the same way along with an as-received sample. The spheroidization observed in the irradiated samples was of the same degree as in the as-received samples. The hardnesses of the as-received sample and the samples that had been irradiated for 3 hr and 9 hr were found to be 23 RC, 22 RC, and 23 RC, respectively, after spheroidizing for 1 hr.

An as-received sample, a sample irradiated for 3 hr and another sample irradiated for 9 hr were polished and sealed in an evacuated Vycor tube. This tube was then heated at 1325°F for 6 hr and air cooled. The degree of coarsness of the spheroids in the as-received sample was observed to be the same as that in the samples that had been irradiated for 3 hr and 9 hr. Their hardnesses were 19 RC, 18 RC, and 18.5 RC, respectively. Thus, radiation damage prior to spheroidization did not seem to enhance the rate of spheroidization.

IV. DISCUSSION OF RESULTS

As discussed in Chapter II, the rate of spheroidization depends on the spheroidizing temperature, which, in turn, depends on the lower critical temperature. The lower critical temperatures of the steels studied were, therefore, determined. The values of the lower critical temperatures obtained by thermal analysis and by heating and quenching were observed to be in very good agreement with those obtained from the literature.

Five different steels were spheroidized by five spheroidizing procedures. Hence, it is rather difficult to discuss all the steels and all the procedures simultaneously. The early part of the discussion to follow, therefore, deals with the features of different procedures independently. In the latter part of the discussion, the response given by individual steels to the different treatments has been reviewed.

A. Spheroidization Treatment:

1. Cold Work Prior to Spheroidization: Although, it is well known that cold work enhances the rate of spheroidization, the idea behind cold working the steels under investigation was to find out the extent to which cold work enhances the rate in these steels. It was observed that the greater the deformation the coarser was the spheroidization. H13 and W1 steels annealed for 8 hr after cold work, Figs. 3b and 6b, were not spheroidized as coarsely as their mill

samples, Fig. 3a and 6a. However, 8 hr of spheroidizing of the other steels after cold work was more satisfactory. Sample M2-5(0.350" dia.), Fig. 5b, looked more coarsely spheroidized than its mill sample but was 5 points harder. On the other hand, sample H13-5(0.350"dia.), Fig. 3b, looked more finely spheroidized than its mill sample but was 2 points harder. The microstructures and hardnesses of A2 and D3 steels after cold work and 8 hr spheroidization, Figs. 2b and 4b, were found to be similar to their respective mill sample microstructures and hardnesses, Figs. 2a and 4a.

In the cold work and annealing process, the maximum furnace time used was 10 hr; 2 hr for softening prior to cold work and 8 hr for actual spheroidization. This total time is approximately one third the time used by the steel mill in their commercial process. Although the spheroidized structures obtained by this procedure were, in general, finer than those in the mill samples, all the hardnesses of the various steels spheroidized for 8 hr after cold work were within the mill hardness specification range for the annealed steels, Table V. It is not known whether some of these finer structures will machine satisfactorily.

The effect of original microstructure prior to cold work was studied with W1 steel because distinctly different microstructures could be produced in this steel easily. Cold working and annealing of the samples that were coarse pearlite did not cause complete spheroidization in 8 hr. The as-received samples that contained fine pearlite produced a dispersion of fine carbide spheroids after cold working and annealing for 8 hr. The coarsest spheroidite structure, Fig. 6d was obtained from a partially spheroidized microstructure. However, a rather long time, 12 hr, had to be spent in achieving the partially spheroidized microstructure prior to the cold work.

2. Cycling Method: In all the steels, the two-cycle samples showed coarser carbides than the single-cycle samples. Two-cycle samples of A2 and D3, Figs. 8a and 8b, were very similar to their respective mill samples both in microstructure and hardness. Their hardnesses were towards the lower side of the hardness specification range for the annealed steels. The two-cycle sample of M2 steel, Fig. 8e, showed spheroids which looked coarser than those in its mill sample. However, its hardness was 5 points higher than its mill sample and 2 points higher than the upper limit of the specification range.

H13 and W1 showed a few very coarse carbides plus fine carbides, Figs. 8d and 8f. A modification was made in the treatment of H13 steel with the intention of dissolving coarse carbides completely. Although this modification resulted in some very coarse carbides, there were others which were still very fine. These fine carbides probably precipitated out afresh on final holding at the spheroidizing temperature. In this modification, it was observed that the austenitizing time did not have an appreciable effect on the hardness values or the microstructures, but the longer holding time at the spheroidizing temperature produced coarser carbides and thus resulted in lower hardness.

The two-cycle samples used 12 hr of furnace time. Their microstructures and hardnesses were comparable with those of the mill samples in most of the steels. However, this method involved handling the steel at high temperatures during the transfer from one furnace to the other. This might not be desirable in commercial heat treatment practice.

3. Isothermal Annealing: The principles involved in isothermal annealing are given by Payson's rules described in Chapter II. As mentioned earlier, these rules apply strictly to straight carbon or low alloy steels. An attempt was made to apply these rules to the highly alloyed tool steels. It was observed that these rules apply to the high alloy tool steels to a large extent. No pearlite was observed in the samples of alloy steels annealed isothermally, but in its place, a dark-etching transformation product was observed in the samples of A2, M2, and D3 steels isothermally annealed for 24 hr. This dark-etching product was apparently rather soft. The same kind of dark-etching product was also observed in the samples transformed at their nose temperatures.

The effect of austenitizing time and temperature in isothermal annealing was studied with W1 steel. All the samples austenitized at 1600°F were pearlitic, an obvious consequence of Payson's first rule which says that the higher the austenitizing temperature, the greater is the tendency of the annealed steel to be lamellar. The spheroids observed in the samples austenitized for 1 hr at 1500°F were almost as coarse as those in the sample austenitized for 2 hr at 1400°F. This is probably because the same degree of homogenity of the austenite was obtained in both these samples. In the sample austenitized for 6 hr at 1400°F, some small colonies of pearlite were observed along with coarse carbide spheroids. This indicated that holding for 6 hr at 1400°F started to homogenize the austenite in some areas. The transformation product in these areas was, therefore, pearlitic. Most of the austenite was nonhomogeneous, however, since it transformed to a spheroidal product.

The sample of A2 steel, isothermally annealed at the spheroidizing temperature, Fig. 10a, had larger massive carbides than the mill sample, Fig. 2a. However, it was harder than the mill sample by 3 points. Isothermal annealing at spheroidizing temperatures gave lower hardnesses and coarser carbides in samples D3-44 and M2-44 than in the mill samples, Figs. 10c and 10d. Although the hardness of sample No. H13-44 was lower than its mill sample by 3 points, on the average, the carbide particle size in H13-44, Fig. 10b, was finer than that in its mill sample.

Maximum furnace time used for isothermal annealing was 33 hr, which is very close to the time used in the commercial annealing process. In view of this fact, rather coarsely spheroidized structures, comparable with the mill sample structures, were not unexpected. Also, the hardnesses of all the samples were either on the lower side or in the middle of the mill hardness specification range for the annealed steels; the only exception being the H13 sample whose hardness was well below the range.

4. Martensite Formation Prior to Spheroidization: Various samples of all the steels were quenched from various temperatures and were subsequently tempered at the spheroidizing temperatures. Samples of each steel were spheroidized to various degrees depending on the austenitizing temperature. Just why a sample quenched from a particular temperature should spheroidize better than others is not clear but it is possible that the homogenity of the austenite or martensite determines the degree of spheroidization on tempering. Thus, at very high temperatures, austenite, or the martensite after quenching, tends to be homogeneous, leaving no carbide or very few carbide nucleii for spheroidization during subsequent tempering. At very low temperatures, austenite, or martensite after tempering, tends to be nonhomogeneous and contains a very fine carbide dispersion. At some intermediate temperature, there might be an optimum number of carbide nucleii which grow coarse enough to give the best spheroidal product.

The hardened and tempered samples, A2-76, H13-76, and D3-76, were harder than their mill samples by 2 to 5 points. Carbides in these samples looked much finer than those in the mill samples. M2-76 showed carbides which were distinct and, more or less, of the same degree of coarseness as those in its mill sample. Both M2-76 and its mill sample had the same hardness.

In general, 8 hr of tempering of these steels produced more finely spheroidized structures than those obtained by other spheroidizing procedures. However, the hardness of all the steels, except H13, tempered for 8 hr were within the mill hardness specification range. H13 sample had a hardness 3 points below the lower limit of the specification range. It is not known whether or not these finely spheroidized steels would machine satisfactorily.

The oil quenched samples of W1 steel were observed to spheroidize more coarsely than the water quenched samples. This may mean that a partially martensitic structure causes coarser spheroidization than a fully martensitic structure, the tempering temperature and time being the same in both cases.

Samples of A2 and D3 quenched from temperatures above 1500°F were observed to have quench cracks. These cracks might have been initiated during cutting of the hot-worked, air cooled, as-received bars. Such quench cracking would, of course, be unacceptable in commercial practice.

5. <u>Radiation Damage Prior to Spheroidization</u>: The effect of irradiation on the precipitation of carbide in a carburized specimen of 99.9% pure "Ferrovac E" iron rod had been studied by Damask(18). He carburized the iron rod at 712°C for 68 hr and quenched it into brine at 0°C. He has

shown that neutron irradiation at 57°C of this quenched specimen increased the rate at which carbon was removed from the supersaturated solid solution of the carburized rod. According to his explanation, two different reactions occur: (i) Carbon atoms are trapped by individual point defects produced by the irradiation, and (ii) irradiation produces nucleation cites for precipitation of the metastable carbide. Similar effects have been observed by Stanley(19) during neutron irradiation of supersaturated Fe-N solutions.

In the present investigation, no nucleation of carbides after irradiation was observed at a magnification of 1000X. Furthermore, the rate of spheroidization was not enhanced by the neutron irradiation. It is possible that the doses of radiation given in the present investigation might have been too small to create any point defects or cause any precipitation of carbide.

It should be realized that this work and the work of Damask and Stanley were different in two respects: (i) The samples were not supersaturated with carbon during irradiation in the present work. Hence, there was no driving force for the precipitation of carbide. (ii) The samples in Damask's investigation were not annealed after irradiation. Precipitation was observed at room temperature in supersaturated samples. Heating of the samples to 1325°F in the present work might have removed most of the effect of the irradiation. Thus, for example, at high temperature, the point defects produced by the irradiation might have been annealed out, thereby eliminating any possible nucleation cites for precipitation.

B. Response of the Individual Steels to the Different Treatments:

1. A2 Steel: The minimum hardness of A2 steel obtained by different spheroidizing procedures was 17 to 18 RC which was 2 to 3 points higher than the mill sample hardness. This minimum hardness was towards the lower side of the hardness specification range.

Table VI indicated that the cold worked samples achieved hardnesses in the specification range only after annealing for 8 hr and furnace cooling. All other cold worked and annealed samples had hardnesses above the range. However, they were all spheroidized. Samples A2-1(0.500" dia.) and A2-2(0.500" dia.) which were not cold worked before annealing showed tempered martensitic structures. The samples annealed for 1 and 2 hr after cold working had extremely fine carbides and such fine carbides might not be acceptable from the machinability point of view.

The coarsest and most uniformly spheroidized structure was given by the cycling method. The carbides in the two-cycle sample, Fig. 8a, looked as coarse or coarser than those in the mill sample but its hardness was 2 points higher than the mill sample hardness. The single-cycle sample was finely spheroidized and its hardness was 2 points higher than the upper limit of the hardness specification range.

The isothermal transformation for 24 and 32 hr followed by furnace cooling resulted in a few very coarse carbides but other carbides were very fine. The hardnesses of these samples were in the middle of the specification range.

The hardened and tempered samples which were furnace cooled had fine carbides. However, their hardnesses were in the specification range. Among all the hardened and tempered samples, sample A2-76, Fig. 12a, that was tempered for 8 hr showed the coarsest carbides. All the samples that were air cooled after tempering showed tempered martensitic structures.

2. <u>H13 Steel</u>: The mill sample of H13 steel had a structure containing rather uniform, coarse carbides. Its hardness was 8 points lower than the lower limit of the specification range. All the H13 samples produced in this study showed carbides that were finer and less uniform in size. The two-cycle sample, H13-22, Fig. 8d, had noticeably coarser carbides than the cold worked and annealed sample, H13-5(0.350" dia.), Fig. 3b. However, H13-5(0.350" dia.) was only 2 points harder than the mill sample, Fig. 3a, whereas H13-22 was 3.5 points harder than the mill sample. Even the single-cycle sample, H13-21, Fig. 8c, was more coarsely spheroidized than the H13-5(0.350" dia.) sample and yet H13-21 was 4.5 points harder. The isothermally annealed

sample, H13-44, Fig. 10b, was softer than the mill sample but the average carbide size in H13-44 seemed to be somewhat finer than that in the mill sample. Apparently, carbide particle size alone does not determine the hardness of the spheroidized material.

The hardness of most of the spheroidized H13 steel samples was either within the specification or much below the range. Even sample H13-1(0.350" dia.) that was spheroidized for only 1 hr after cold work achieved a hardness of 18 RC which is the upper limit of the range. This sample was spheroidized but the carbides in this sample were extremely fine. Thus, it was observed that low hardness could be readily achieved in H13 steel, but it was difficult to achieve coarsely spheroidized structures.

3. D3 Steel: The carbides in D3 steel were of two different kinds. Massive carbides were observed in a matrix containing finely dispersed carbides. The finer carbides were of rather uniform size as were the massive carbides in most of the D3 samples. Thus, the presence of two different carbide sizes was not considered to be an indication of "nonuniformity". In this respect D3 was generally more uniform than H13 and W1, both of which had a range of carbide sizes.

D3 steel seemed to spheroidize readily, and, except for the high temperature tempering, all the methods gave well spheroidized structures that were comparable to the structure of the mill sample. The softest samples obtained by the different procedures had hardnesses only 1 to 2 points different from that of the mill sample. In particular, the two-cycle sample, Fig. 8b, was softer than the mill sample by one point and the carbides in this sample were very distinct and somewhat coarser than those in the mill sample.

Most of the cold worked and annealed samples and the quenched and tempered samples had hardnesses above the specification range. In fact, the only samples that had hardnesses lower than the upper limit of the range were D3-5(0.350" dia.), D3-5(0.397" dia.) and D3-76. Hardnesses of both single and double cycle samples as well as all the isothermally annealed samples were within the specification range. Thus, D3 steel responded better to the cycling method and isothermal annealing than to cold working and annealing and high temperature tempering.

4. M2 Steel: All the cold worked and annealed samples and both the single and the double cycle samples of M2 steel were spheroidized but none of these samples could meet the hardness specifications for annealed M2 steel. The samples isothermally annealed for 24 and 32 hr and furnace cooled were coarsely spheroidized and their hardnesses, Table XI, were in the specification range. The hardnesses of the samples tempered for 8 hr, Table XVI, could meet the specifications but those tempered for 2 and 4 hr were too hard and were more finely spheroidized.

The cold worked and annealed sample, M2-5(0.350" dia.), Fig. 5b, the two-cycle sample, M2-22, Fig. 8e, and the tempered sample, M2-76, Fig. 12d, had very similar microstructures and resembled that of the mill sample. However, the first two samples were five points harder than the mill sample while the tempered sample had the same hardness as the mill sample. M2 steel responded very well to the isothermal annealing. The hardness of the isothermally annealed sample, M2-44, was lower than that of the mill sample by one point. The carbides in M2-44, Fig. 10d, looked much coarser than in the mill sample.

5. <u>W1 Steel</u>: The mill sample of W1 steel differed from other W1 samples in microstructure in that the carbides in the mill sample were more rounded, uniform and somewhat coarser than those in the samples produced in this study. All the procedures worked equally well on this steel and the softest samples obtained by the different procedures were about 1 to 4 points harder than the mill sample.

Except for sample W1-5(0.350" dia.), all the as-received samples which were cold worked and annealed had hardnesses above the specification range. Other than this group of samples, all the samples of W1 steel met the hardness specifications. Most coarsely spheroidized but rather nonuniform carbides were shown by the two-cycle sample, Fig. 8f. Its hardness was the same as the lower limit of the specification range.

V. CONCLUSION

1) A2 steel spheroidized very well by different procedures but the hardness of the spheroidized samples was never as low as the mill sample hardness. This steel responded the best to the cycling method.

2) Low hardness could be readily achieved in H13 steel, but it was difficult to achieve coarsely spheroidized structures. The softest and the most coarsely spheroidized structure in this steel was given by isothermal annealing.

3) D3 steel spheroidized readily. Also, most of the samples had hardnesses that were within the specification range for annealed D3 steel. In general, the cycling method and isothermal annealing worked better than cold working and annealing or high temperature tempering.

4) All the procedures gave well spheroidized structures in M2 steel. But only isothermal annealing gave hardness lower than that of the mill sample. The carbides in the isothermally annealed sample were much coarser than in the mill sample.

5) All the procedures worked equally well on W1 steel. However, none of the procedures gave as rounded, uniform, and coarse carbide structure as that of the mill sample. The most coarsely spheroidized structure with rather nonuniform carbides was shown by the two-cycle sample.

6) Although the cycling method involved handling the steel at high temperature, it used only 12 hr of furnace time compared to 32 hr required for the isothermal annealing. In this respect, the cycling method for A2, D3, and W1 steels might be more economical than isothermal annealing. Isothermal annealing is similar to the slow cooling procedure used at the mill and it uses approximately the same amount of furnace time.

7) Irradiation did not enhance the rate of spheroidization in W1 steel.

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VII. VITA

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