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# The effect of temper carbon nodule number on the physical properties of a malleable iron

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Department:

# THE EFFECT OF TEMPER CARBON NODULE NUMBER ON THE PHYSICAL PROPERTIES OF A MALLEABLE IRON

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A

THESIS

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## INTRODUCTION

It is known that the number of temper carbon nodules per unit volume of malleable iron has an effect on both the rate of anneal and on the resulting physical properties, but neither have been clearly worked out quantitatively. The purpose of this investigation is to ascertain the effect of temper carbon nodule number on certain of the physical properties of a commercial malleable iron.

Quantitative determinations of the effect of temper carbon nodule number would be essential from a theoretical viewpoint in working out the mechanism of graphitization, and would be useful in commercial practice in showing the most desirable nodule count and matrix structure to have in order to produce optimum mechanical and physical properties. The metallurgical potentialities of malleable iron will not be fully realized until the graphite formation during the annealing cycle and the resulting physical properties due to this formation are fully understood and controlled.

## MALLEABLE CAST IRON DEFINED

The general term "cast iron" includes grey irons, pig irons, white cast irons, chilled (white face) cast irons, malleable irons, and the new inoculated nodule irons. Cast irons are essentially alloys of iron, carbon, and silicon in which the carbon is present in excess of the amount which can be retained in solid solution in austenite at the eutectic temperature.

Grey iron — Cast iron which contains a relatively large

percentage of its carbon in the form of flake or lamellar graphite, and substantially all of the remainder of the carbon in the form of eutectoid carbide.

White iron -- Cast iron in which substantially all of the carbon is present in the form of iron carbide.

Malleable iron -- Essentially an alloy of iron, carbon and silicon, such that the "as cast" structure is white iron, but after being cast is converted structurally by heat treatment into a matrix of ferrite containing nodules or spheroids of temper carbon, (graphite).

Pearlitic malleable iron -- Differs according to the American Society for Testing Materials from standard malleable in that the matrix contains varying amounts of pearlite replacing the ferrite.

Graphitic carbon (in a steel matrix) is ordinarily distributed in two basic ways, the first as lamellar or flake graphite, the second as nodules or spheroidal graphite. The basic forms may vary in size, both flake and nodule are found in a finely divided form referred to as [1] supercooled, reticular, and dendritic.

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(1) Morrough, H. and Williams, W.J., Graphite Formation in Cast Iron, Engineering, Aug. 8, 1947, p. 141.

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The optical anisotropy of graphite flakes and nodules can be studied by the polarizing microscope. [2]

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(2) Ibid.

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The color of a graphite crystallite will vary twice from light brown to black as the specimen is rotated through 360 degrees.

Owing to this reflex pleochroism it has been possible to show that many graphite flakes have complex needle-like structures internally and are usually aggregates of interlacing crystals. Graphite spherulites consist of radially oriented crystallites growing from a common center (3), (4) with the radii of the

(3) Ibid.

(4) Strauss, H.E., Structure of Spherulites in Nodular Cast Iron, J. of Metals, p. 249, March 1951.

nodules at (0001) direction normal to the basal planes of graphite. Regardless of the basic form it has been reported that the unit cell crystal structure is approximately similar. Graphite (5) may be considered a matrix of two closely related structures, Alpha and Beta. (See illustration 1, page 4.)

(5) Owen, W.S. and Street, B.G., The Crystal Structure of Graphite in Cast Iron, J. of the Iron and Steel Inst., p. 113, Feb. 1951.

The α-graphite structure, shown in Fig. 1a, in which the atoms are arranged in layers (within which they form a regular pattern of hexagons) such that neighboring layers are displaced, as shown in Fig. 1b, and alternate layers lie directly beneath each other. This structure can be described in terms of a hexagonal lattice, space group $D_{6h}^4$, with four atoms in the unit cell. The dimensions of the unit cell are given by Trzebiatowski as $a = 2.456$ Å., $c = 6.694$ Å.

The β-graphite structure, in which two layers are arranged as in Fig. 1b but the next layer is displaced by an equal amount in the opposite direction, as shown in Fig. 1c,

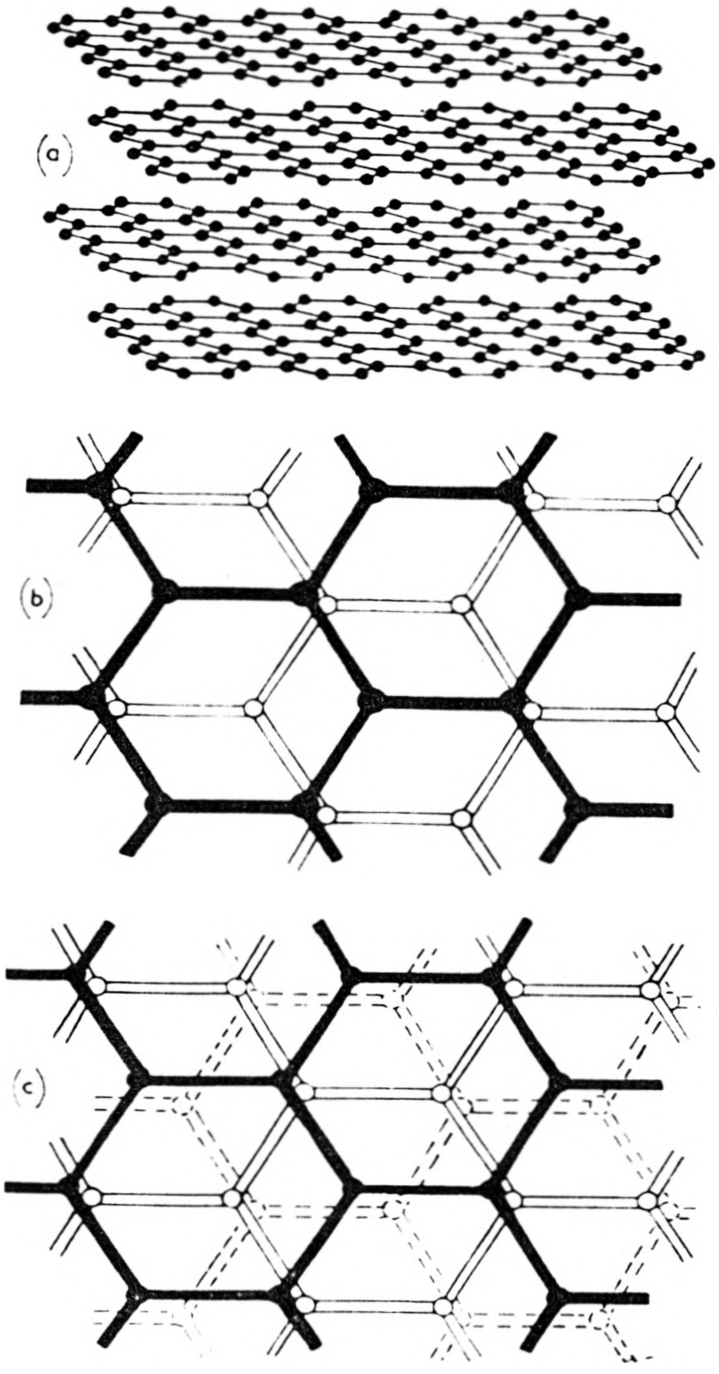


Fig. 1—Structure of α- and β-graphite. (*a*) α-graphite. (*b*) Method of packing layers in α-graphite. The first layer is represented by heavy lines, and the second layer by thin lines. (*c*) Method of packing layers in β-graphite. The first layer is represented by heavy lines, the second by thin lines, and the third by dotted lines.

the pattern being repeated at every fourth layer. This structure can be described in terms of a hexagonal lattice with a unit cell of size length c equal to $\frac{3}{2}$ times that in the alpha-structure, and it has a space group $D_{3d}^{5}$ with unit cell dimensions a = 2.256 Å. and c = 10.044 Å., containing six atoms at co-ordinates 0 0 0, $\frac{1}{3}\ \frac{2}{3}$ 0, 0 0 $\frac{1}{3}$, $\frac{2}{3}\ \frac{1}{3}\ \frac{1}{3}$, $\frac{1}{3}\ \frac{2}{3}\ \frac{2}{3}$, $\frac{2}{3}\ \frac{1}{3}\ \frac{2}{3}$ .

This structure is really rhombohedral, with a = 3.636 Å., alpha = 39.49°, and the atoms at ± (u u u), where u = $\sqrt{\frac{1}{6}}$. The space group is $R\bar{3}m$. The parameter u cannot be determined exactly, but the value of $\frac{1}{6}$ was chosen because it gave plane hexagonal rings.

Owen and Street present nine forms of graphite and their percentage of Beta structure ---

| Type | % Beta-structure |
|---|---|
| 1. natural graphite (Ceylon) | 13.0 |
| 2. hi-purity iron (C-Si alloy) | 7.0 |
| 3. commercial hi-phos grey iron | 7.4 |
| 4. hi-grade grey iron (not inoculated) | 6.6 |
| 5. iron inoculated with SMZ (SMZ - 60% Si and Mn, Zr) | 6.3 |
| 6. malleablized white iron (flake aggregate) | 7.4 |
| 7. malleablized white iron (spherulitic) | 3.5 |
| 8. Cerium treated nodular iron (1) | 6.3 |
| 9. Cerium treated nodular iron (2) | 4.7 |

From this study, they have stated that -- the crystal structure of any particular microscopic form of graphite is independent of the composition and method of manufacture of the iron

the regularity of the packing of the atom layers in graphites occurring in flake form is different from that in spherulitic variety, the flake having less disordered structures and more beta-structure than the spherulitic; the graphite present in iron alloys does not contain any element, other than carbon, in any appreciable quantities. This last statement is under debate at present both in the United States and in England. (6) (7)

(6) Schwartz, H.A., Blackheart Malleable Iron, American Foundryman, p. 46, June 1949.

(7) Morrough, H., op. cit., p. 2.

The strength of graphite has been reported as being approximately 2000 psi. (8) It is felt that the volumes occupied by graphite in the steel matrix of both grey, malleable and nodular irons are stress concentrators (with reference to malleable and nodular irons) (9), and planes of weakness in the

(8) United States Atomic Energy Commission Paper, Shear and Tensile Strength of Graphite Anodes, Clinton Lab.

(9) Flinn, R.A., (Private communication) unpublished material from American Brake Shoe Research Laboratories.

case of grey iron. The important function of the graphite is its shape, size, and distribution, for it will be these properties that will exert the greatest influence upon the matrix structure.

Essentially then, the scope of this investigation will cover an attempt to pre-treat white cast iron to cause variation in the temper carbon nodule number; followed by subjecting this material to a commercial annealing treatment; and subsequently observing the resulting metallographic structure and physical properties.

The physical and mechanical properties to be investigated are: Tensile strength, Yield strength, Elongation, Reduction of area, Brinell hardness, Tukon hardness of matrix structure, Charpy impact strength, and Coercive force.

## REVIEW OF THE LITERATURE

In 1931 the American Society for Testing Materials held a symposium on malleable iron castings. (10) At that time it was reported, that the average values of tensile strength, yield strength and elongation of malleable iron, determined from the results of twenty thousand tests by seventeen investigators were as follows:

| | |
|---|---|
| Tensile Strength | 54,000 psi |
| Yield Strength | 36,000 psi |
| Elongation in 2 in. | 18% |
| BHN | 100-140 |

These values were for what is now considered standard malleable iron, or malleable iron produced by air furnace, open hearth or electric furnace process, or one of the duplexing processes.

Today the ASTM has set up standards on three main types of malleable iron; standard malleable, cupola malleable, and pearlitic malleable. Each type has a distinct set of specifications.

STANDARD MALLEABLE (ASTM A 47-48)

| | Type 32510 | Type 35018 |
|---|---|---|
| Tensile strength | 50,000 psi | 53,000 psi |
| Yield strength | 32,500 psi | 35,000 psi |
| Elongation in 2 in. | 10% | 18% |

CUPOLA MALLEABLE (ASTM A 197-47)

| | |
|---|---|
| Tensile strength | 40,000 psi |
| Yield strength | 30,000 psi |
| Elongation in 2 in. | 5% |

(10) Symposium on Malleable Iron Castings, ASTM, Vol. 31, p. 317, 1931.

PEARLITIC MALLEABLE (ASTM A 220-50T)

| | Type 43010 | 48005 | 53004 | 60003 | 70002 |
|---|---|---|---|---|---|
| T S (psi) | 60,000 | 70,000 | 80,000 | 80,000 | 90,000 |
| Y S (psi) | 43,000 | 48,000 | 53,000 | 60,000 | 70,000 |
| Elong. (%) | 10 | 5 | 4 | 3 | 2 |
| BHN | 163-207 | 179-228 | 197-241 | 197-241 | 241-285 |

The ASTM does not specify matrix structure, or temper carbon nodule number. Schwartz [11], Schindler, and Elliott

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(11) Schwartz, H.A., Schindler, H.J., and Elliot , J.F., The Relation of Carbon Nodule Size and Tensile Properties of Malleable Cast Iron, Proc. ASTM, Vol. 39, p. 583, 1939.

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in 1938 were the first to publish data relating tensile strength to temper carbon nodule count. In their work 16 tensile test bars were divided into four groups, each group subjected to a different heat treatment as follows:

Gp. A was heated to 950 C (1742 F) for one hour, oil quenched and drawn for about a half hour at 500 C

Gp. B was similarly heat treated except that the initial temperature was 825 C (1515 F)

Gp. C was given no heat treatment, but was used as cast

Gp. D was packed in a three inch pipe in pig iron borings, heated for 26 days at 725 C, cooled to room temperature and heated for 22 days more at 725 C, cooled to room temperature and heated 25 days at 710 C. The interruptions were made to permit micro examination of the specimens to learn the progress of graphitization.

Groups A, B, and C were then given a commercial anneal. The specimens were then turned to 0.505 inches diameter with 3/4 inch threaded ends.

The yield strength reported is the Johnson's elastic

limit, where the relation of strain to stress has become one and one half times the initial relationship.

Their results were as follows:

| Gp. | T S (psi) | Y S (psi) | Elong. (%) | R A (%) | Nodules per $mm^3$ | per $mm^2$ approx. |
|---|---|---|---|---|---|---|
| A | 53,940 | 29,000 | 16.5 | 14.2 | 40,686 | 200 |
| B | 54,885 | 27,325 | 11.9 | 9.7 | 28,456 | |
| C | 53,255 | 30,175 | 21.7 | 19.4 | 135 | |
| D | 56,550 | 37,375 | 21.3 | 20.8 | 30 | 5 or 6 |

It appears that the tensile strength is practically not effected by the distribution of the temper carbon except in the case of group D. Since, in order to produce very coarse nodules, the anneal was conducted entirely below the critical point, it is barely possible, but not likely, that the difference is due to the heat treatment of the ferrite. Some hardness readings would have been helpful, but none appear in this paper. The elongation of the relatively coarse nodule iron is definitely higher than that containing the fine nodules. The reduction of area follows a similar rule. Schwartz's conclusions were: "The nodule size of the temper carbon has little effect on either the yield or tensile strengths of malleable iron as determined on machined specimens. A decrease in nodule number from about 28,000 to 135 per cubic mm. is accompanied by an important improvement in ductility. No sufficient theory exists with regard to the physical properties of heterogeneous mixtures to permit rational explanation. It is unfortunate that so large a gap exists between the nodule numbers for groups B and C. This is due to the fact that while prequenching treatment

will give very fine nodules [12] no method was known to us for insuring that the present metal could be annealed with

(12) Schwartz, H.A., Johnson, H.H., and Junge, C.H., Graphitization of Prequenched White Iron, Trans ASM, Vol. 17, p. 383, 1930.

a nodule number of say 1000 or 2000 per cubic mm."

A further check through the literature available to the writer yields no further published American work.

There is however a Russian symposium by S. S. Saltykov [13]

(13) Saltykov, S.S., Role of Center of Graphitization in the Annealing of Malleable Iron, Metallurg., Vol. 14, pp 10-22, 1939.

which considers the influence of the number of centers of graphitization (temper carbon nodules) upon the mechanical properties of malleable iron. In this work the quenching medium is oil, and the results are reported as follows:

| Nodules per $mm^2$ | Tensile Strength | Elongation | Ferrite grain dia. (microns) |
|---|---|---|---|
| 20 to 50 | 49,600 | 12.9 | 30-60 |
| 1200 to 2200 | 52,900 | 6.3 | 20-30 |

As a result of his work Saltykov believes there must be an optimum number of graphite particles between 120 and 1000 per square mm., which will yield maximum physical properties. He also notes the increase in tensile strength with increase of temper carbon nodule count, and the corresponding decrease in elongation. It is also stated that the impact strength of these specimens did not differ to any noticeable extent.

Another review article by M. Gabriel Joly [14] also con-

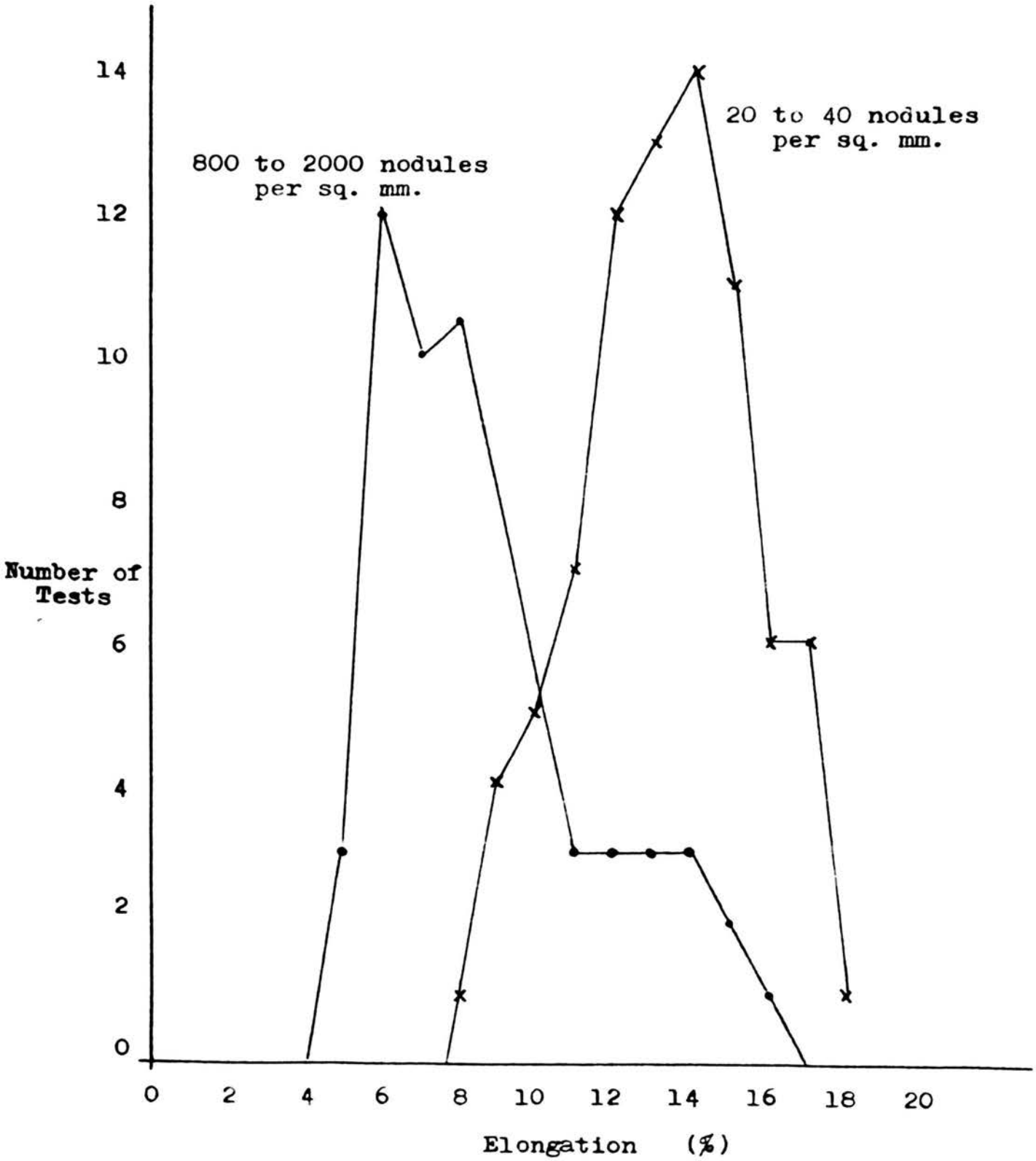
(14) Joly, M. Gabriel, Centers of Crystallization of Carbon in the Annealing of Black Heart Malleable Iron, Fonderie, p. 375, Nov. 1946.

siders the influence of the number of centers of crystallization (temper carbon nodule number) upon the mechanical properties. In Joly's series of tests one set of specimens was subjected to what he terms an oil temper near 930 C (1710 F) before the anneal. This resulted in specimens which had a temper carbon nodule count between 800 and 2000 per $mm^2$. Another set of specimens were annealed as cast, and contained between 20 and 40 temper carbon nodules per $mm^2$. Each set of specimens was subjected to tension until rupture. He then plotted the elongation and tensile strength values of the two series. (See graph 1, page 13, and graph 2, page 14.) From these graphs we may compare the relative frequency of the elongation and tensile strengths values for each series. It is evident that as the temper carbon nodule count increases, <u>generally</u> the elongation decreases, and the tensile strength increases.

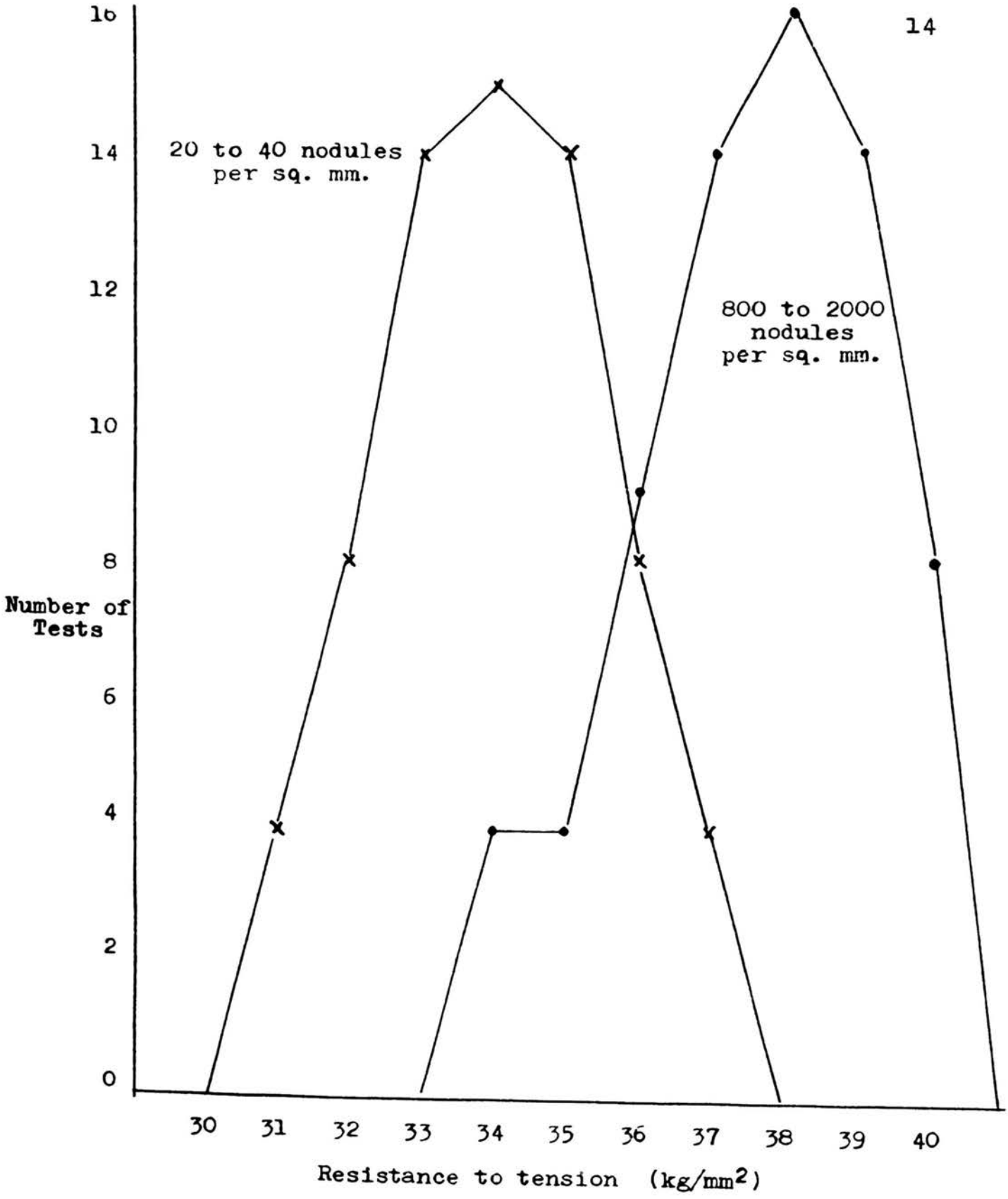
Joly found that the approximate values were true --

| Nodules per $mm^2$ | Tensile Strength (psi) | Elongation (%) |
|---|---|---|
| 1100 | 56,000 | 6 |
| 600 | 52,600 | 10 |
| 150 | 50,000 | 12.5 |

He states: "It should be noted that one does not observe great variation in the mechanical characteristics when there is a large variation in the number of temper carbon nodules. Two irons, having for example 30 and 120 centers per $mm^2$ have




GRAPH 1 - Influence of the number of nodules per sq. mm. on the elongation of a malleable cast iron.




GRAPH 2 - Influence of the number of nodules per sq. mm. on the resistance to tension (tensile strength)

almost the same mechanical properties." Joly considers a malleable iron with from 60 to 140 centers of crystallization carbon (temper carbon nodule number) as one that will yield optimum mechanical properties.

The only other reference found was in A. Hultgren and O. Edstrom's article on graphitization of martensite [15],

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(15) Hultgren, A., and Edstrom, O., Graphitization of Martensite on Heating, Jernkontorets nnaler, Vol. 26, p. 83, 1942.

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in which it was noted that -- Tensile tests on prequenched and malleablized specimens showed a normal tensile strenth, but poor elongation (6-7%). The tensile strength was lower for alloyed irons than for un-alloyed irons. They gave no data, or offered any explanation for this statement.

## DISCUSSION OF PRESENT STUDY

**Material Used:** The material used was in the form of standard tension test specimens (as cast - 7 1/2" long, 5/8" in the smallest section) as specified in the ASTM A-47-48, Standard Specification for malleable iron castings, Section 5.

The bars were obtained from Chicago Malleable Castings Company. Frank A. Czapski, Chief Metallurgist of the company has furnished the following information concerning them. They were molded in green sand, pattern faced with a fine grain synthetic sand to produce a fine surface finish on the test bars. The molds were poured from hand ladles, taken from one 800 lb. transfer ladle. An addition of 0.001 % B (Boron) was made to this transfer ladle. Their lab report of the chemical analysis of the test bars is --

| | |
|---|---|
| C | 2.49 % |
| $M_n$ | 0.05 |
| Si | 1.13 |
| P | 0.13 |
| S | 0.14 |
| $C_r$ | 0.048 |

The general melting procedure was as follows: a # 7 cupola lined to 48 inches; then to a reverberatory holding furnace, fired with pulverized coal. Average melting rate 12 tons/hr. Cupola charge --

| | | |
|---|---|---|
| metal | 2000 lb. | 7% malleable scrap or foundry pig |
| | | 5% silvery pig (15%) |
| | | 50% white iron sprue |
| | | 38% steel scrap |
| coke | 230 lb. | |
| Limestone | 65 lb. | |
| Purite | 2 lb. | |

Any deficiency of manganese content in charge is com-

pensated by the addition of Mn-Si briquettes.

Approximate analysis of composite charge --

| | |
|---|---|
| C | 1.56 % |
| Mn | 0.65 |
| Si | 1.45 |
| P | 0.12 |
| S | 0.15 |

The approximate analysis of metal leaving the cupola --

| | |
|---|---|
| C | 2.5-2.7 |
| Mn | 0.5-0.55 |
| Si | 1.1-1.3 |

They try to maintain the following chemistry range in the duplex iron --

| | |
|---|---|
| C | 2.4-2.5 |
| Mn | 0.45-0.5 |
| Si | 1.1-1.2 |
| P | 0.1-0.14 |
| S | 0.14-0.16 |

The annealing cycle used by Chicago Malleable Castings Company is --

| | | |
|---|---|---|
| 36 hr. | to | 1600 F |
| 60 | at | 1600 F |
| 30 | to | 1280 - 1320 F |
| 24 | at | 1280 - 1320 F |
| 24 | to | 1150 F |
| open | at | 1150 |
| 174 hours total | | |

The annealing furnaces are fired with pulverized coal, and the castings are packed in gravel. It should be noted that this cycle is rather long, however, it is satisfactory for the heavy type of castings produced in this plant.

## EXPERIMENTAL METHODS

It is known that the following factors (16)to(26) effect the

(16) Saltykov, S.S., op. cit., p. 11.

(17) Joly, M. Gabriel, op. cit., p. 12.

(18) Hultgren, A., and Edstrom, O., op. cit., p. 15.

(19) Schwartz, H.A., Solved and Unsolved Metallurgical Problems of Blackheart Malleable Iron, Proc. Inst. of British Foundrymen, paper # 897, 1947-48.

(20) Palmer, S.W., The Anneability of White Iron in the Manufacture of Malleable Iron, Foundry Trade Journal, p. 110, Oct. 9, 1947, p. 129, Oct. 16, 1947.

(21) Elsea, A., and Lorig, C.H., The Effects of Copper Content and Low Temperature Pretreatment of Some White Irons on Malleablization, Trans., AFS, p. 1032, 1943.

(22) Palmer, S.W., The Influence of Heating Rate in Malleable Iron Annealing, Paper # 931, Proc. Inst. of British Foundrymen, p. A-40, 1949.

(23) Hall, H.G., Graphitization in the Malleable Iron Process, Paper # 961, Proc. Inst. of British Foundrymen, p. A-146, 1949.

(24) Schwartz, H.A., Alloying Elements Effect on Tensile Properties of Malleable Iron, Amer. Foundrymen 13, # 4, 130-3, 1948.

(25) Simmons, O.W., Quenching Rate Versus Graphite Formation in Prequenched White Cast Iron, ASM preprint # 29, 1943.

(26) Ohamoto, Dr. M., Effect of Heating Rate on Graphitization of White Iron, Foundry, Feb. 1951.

resulting temper carbon nodule number --

a - the basic chemistry and metallurgical history of the pig iron used
b - type of melting and holding furnaces
c - superheating of the white iron
d - ddition of alloying elements to the basic white iron
e - basic chemical composition of the white iron
f - rate of solidification of the white iron
g - thermal pretreatment before annealing
h - cold work before annealing
i - annealing atmosphere
j - rate of heating to second stage holding temperature
k - annealing temperature

It is evident that in our investigation the factors (a) through (f) are constants, since the metal was poured from a single heat and ladle into identical molds. There is then a choice of factors (g) through (k). Of these, the thermal pretreatment, and mechanical work before annealing are the only factors that will allow a large range of uniform temper carbon nodule variation. Mechanical cold work causes too much variation within the same specimen, the nodule count varying directly with the stress concentration. Annealing atmosphere, rate of heating to second stage, and

annealing temperature will cause some variation in nodule count, but their main effect is in the variation of shape of the temper carbon nodule.

Pre-treatment: Fourteen different thermal treatments were selected. (See Table A, page 21). The thermal treatments will be referred to as Groups 1 to 14, and will consist of three tensile test bars each.

The thermal treatments were selected as such, in an attempt to obtain temper carbon nodule counts between 50 and 1000 per $mm^2$.

The heat treatment was performed using a "Hayes Electric Glo-bar furnace", temperature fixed by a Bristol Controller. During the heating the furnace was flooded with commercial nitrogen to lessen the chance of surface oxidization of the test bars.

White iron hardness readings: After the white iron bars were heat-treated, a flat was ground on the 3/4 inch face. The piece was then lightly ground on the opposite side to remove any trace of glaze that might have been caused by quenching. Rockwell C hardness readings were made on all bars.

Annealing cycle: White cast iron at room temperature consists of pearlite and cementite. When white cast iron is heated above the ($A_1$) critical point, pearlite changes to austentite, which is capable of dissolving increasing amounts of cementite at increasing temperatures. For a definite temperature and time, a saturation point is reached, and the structure will consist of saturated austentite and free cementite.

## TABLE A

Table of thermal treatments to which the test bars were subjected before annealing.

| GROUP | TREATMENT |
|---|---|
| 1. | as cast (three bars each). |
| 2. | heat to 800 F in lead, hold for 16 hr., air cool. |
| 3. | heat to 1500 F, hold at temperature 15 min., furnace cool. |
| 4. | heat to 1500 F, hold at temperature 15 min., air cool. |
| 5. | heat to 1500 F, hold at temperature 15 min., quench in air stream. |
| 6. | heat to 1500 F, hold at temperature 15 min., quench in water at 212 F. |
| 7. | heat to 150) F, hold at temperature 15 min., quench in water at 120 F. |
| 8. | heat to 1500 F, hold at temperature 15 min., quench in water at 65 F. |
| 9. | heat to 1500 F, hold at temperature 15 min., quench in salt solution (NaCl - 10%) at 65 F. |
| 10. | heat to 1700 F, hold at temperature 15 min., quench in water at 212 F. |
| 11. | heat to 1600 F, hold at temperature 15 min., quench in water at 212 F. |
| 12. | heat to 1360 F, hold at temperature 15 min., quench in water at 212 F. |
| 13. | heat to 1200 F, hold at temperature 15 min., quench in water at 212 F. |
| 14. | heat to 1500 F, hold at temperature 15 min., quench in oil at 70 F. (Haughton # 2, soluble). |

Assume a temperature of 1700 F, the structure will consist of austenite and cementite, and as this temperature is maintained, cementite will dissolve in austenite. While this is going on and before equilibrium is reached, the less soluble carbon or free graphite is being precipitated from the austenite. As annealing continues, more carbon is precipitated from the austenite as free graphite, the nodules of which rapidly increase in size.

During precipitation the austenite absorbs free cementite to maintain its saturation or equilibrium which is continually tending to be lowered by the precipitation of carbon as graphite. This continual precipitation of graphite and corresponding absorption of cementite, continues until there is no free cementite left. Equilibrium, for the temperature chosen, has been attained and the metal consists entirely of saturated austenite and free graphitic carbon.

In theory there is no advantage in holding at this temperature (1700 F) after equilibrium is reached. Since austenite cannot hold as much carbon in solution at lower temperatures, it will be necessary to lower the temperature below 1700 F in order to precipitate more graphite. At the critical range, around 1328 to 1333 F, austenite can hold in solution only about 0.6% graphitic carbon, the equivalent of about 9% cementite; where as just short of 2066°F austenite can dissolve 25.5% cementite. Therefore the metal is allowed to cool slowly to the $A_1$, (1328 F approximately) graphitic carbon being progressively precipitated

on to the existing nodules, and the austenite becoming lower and lower in carbon content. At just below the $A_1$, roughly 0.6% carbon remains in the solution of austenite, the remainder of the carbon being present as graphite nodules. If the temperature is dropped too fast at this stage the 0.6% carbon is retained in the metal as pearlite. However, if the metal is held for some time at 1325 and to 1330 F, then allowed to cool slowly to approximately 1250 F most of the eutectoid carbon will precipitate as graphite, and a matrix structure of ferrite will result.

For this investigation the annealing cycle will be divided into five stages as follows --

| | |
|---|---|
| 1st stage | heat up period to annealing temperature |
| 2nd stage | holding temperature |
| 3rd stage | slow cool to 1330 F (10 F/hr.) |
| 4th stage | hold at 1325 to 1333 F |
| 5th stage | cooling to room temperature |

In order to ascertain the time for stages one and two, dilatometric studies were made on two inch specimens cut from the 3/4 inch section of a test bar from groups 3 and 9. Group 3 being the annealed specimens, and group 9 the salt solution quench at room temperature. These groups should represent the extremities in annealing rates.

The basis for these studies is that as the cementite of a white iron decomposes during graphitization, an expansion of the white iron occurs. This expansion is caused by the graphite, having relatively low density, making room for itself in a metallic matrix of considerably higher density. (27)

(27) Palmer, S.W., The Anneability of White Iron in the Manufacture of Malleable Iron, op. cit. p. 18.

As a result of these tests conducted at 1700 F it was found that the test bar from group three required six hours to reach a period of stable size, and the one from group nine required three hours. (See graphs No. 3 and 4, pages 25 and 26.)

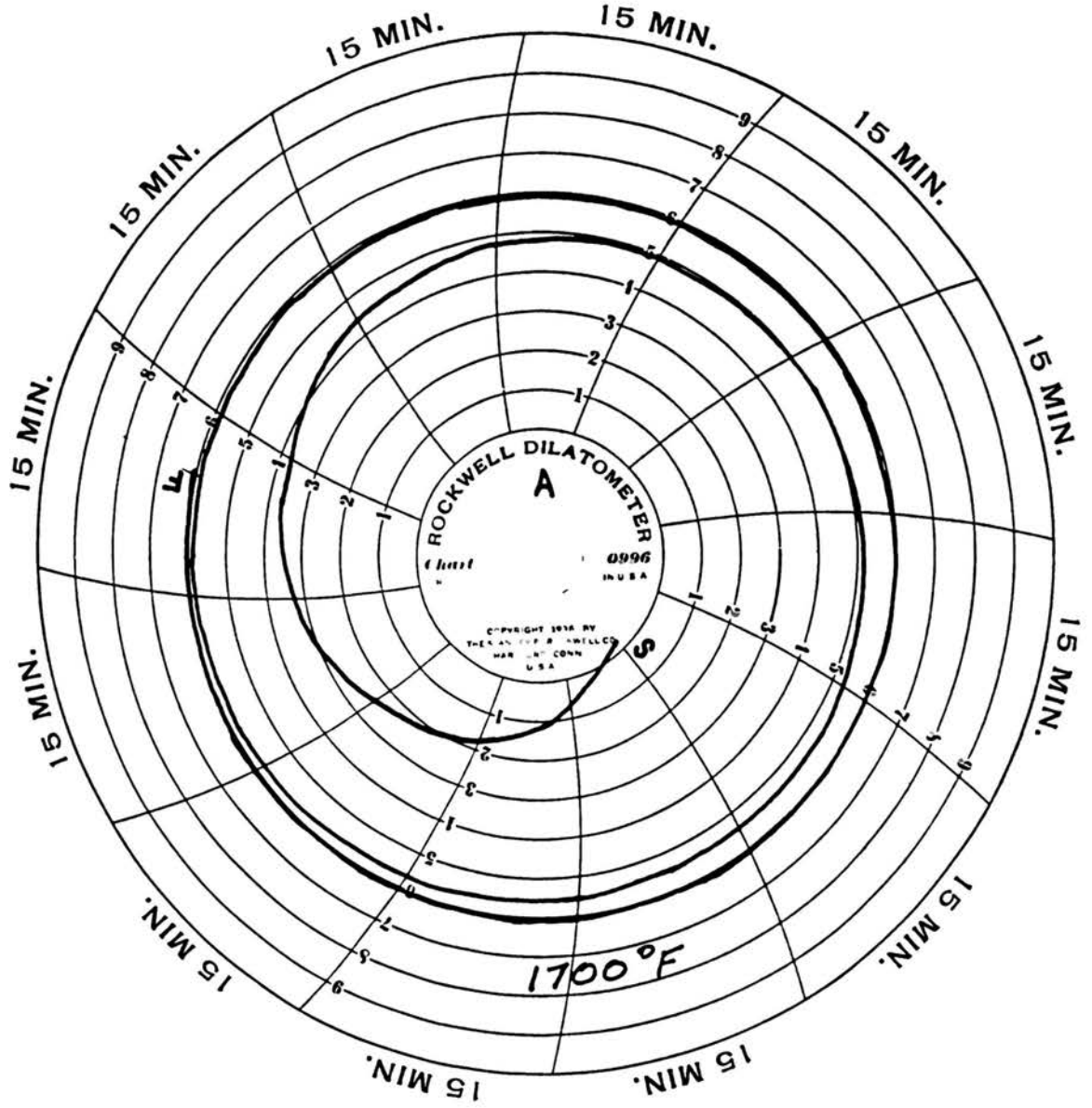
The third and fourth stages have somewhat been standardized within the industry. The American Malleable Iron handbook recommends a rate of 10 F per hour or less.

After the temperature is below the $A_1$ the rate of cooling is of no consequence. Hence stage five is variable.

The annealing cycle used in this investigation is presented in graphic form (see graph No. 5, page 27) and is as follows:

| | | |
|---|---|---|
| 10 hrs. | to | 1700 F |
| 20 hrs. | at | 1700 F |
| 2 hrs. | to | 1400 F |
| 6 hrs. | to | 1340 F |
| 6 hrs. | at | 1340 F |
| 6 hrs. | to | 1275 F |
| open furnace and cool to room temperature | | |
| 50 hrs. | | |

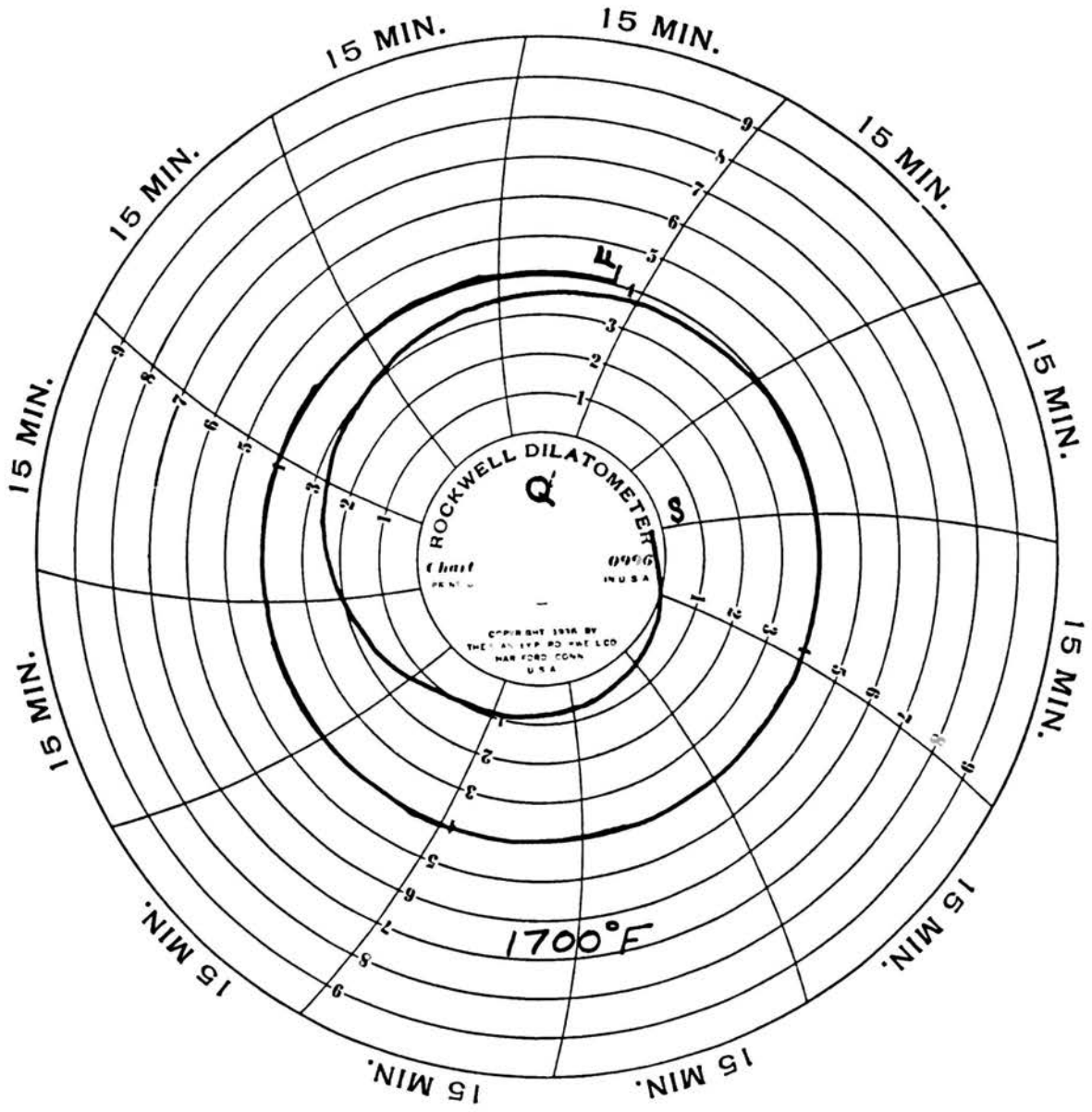
Due to the small cross section of the specimens, and the 1700 F annealing temperature it is believed the specimens will fully anneal in this time period. Mr. Czapski of Chicago Malleable was consulted concerning this matter and believed that the 50 hour cycle will probably completely graphitize the structure of the test bars.




GRAPH 3 - Dilatometric study of 1st and 2nd stage graphitization of test specimen from Group 3

Temperature 1700 F

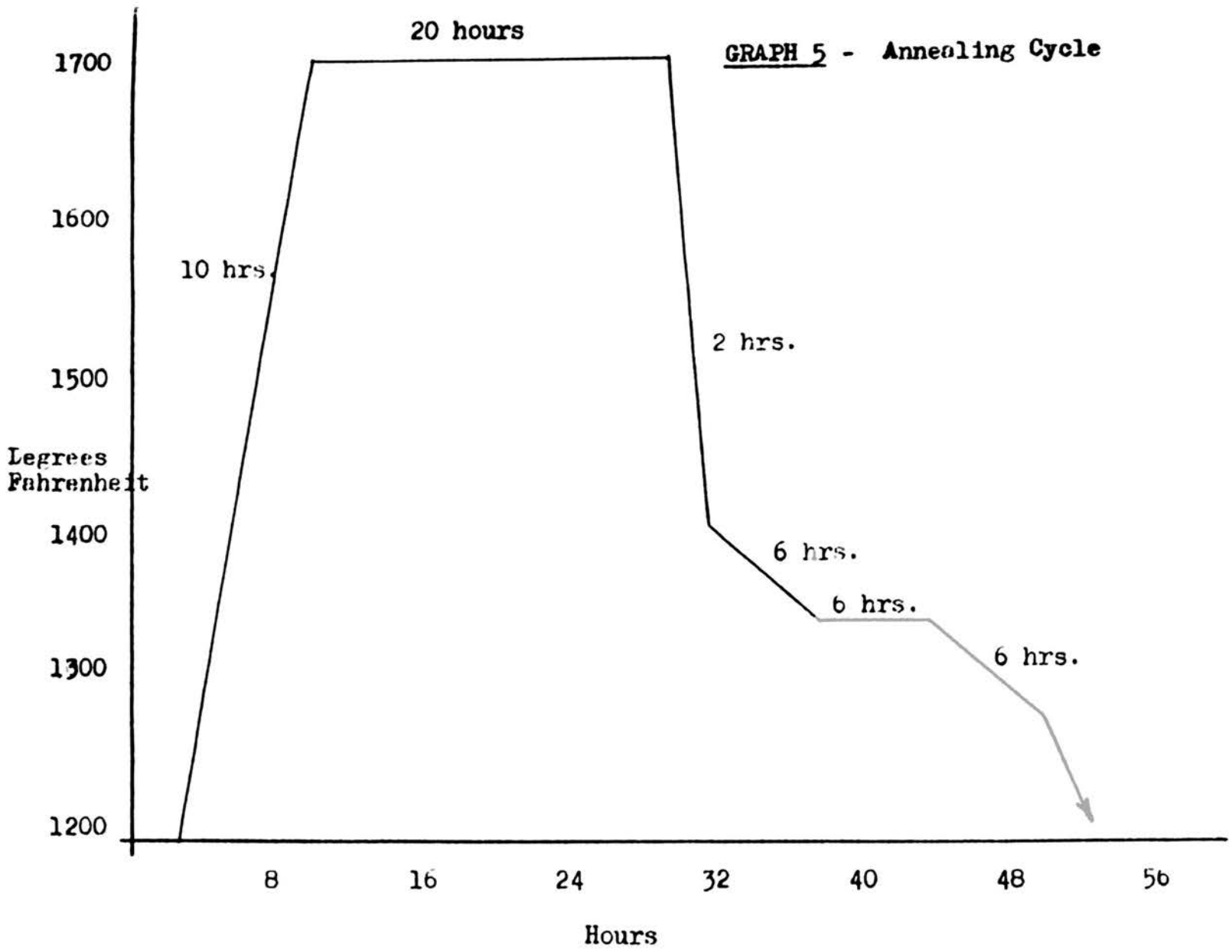
Time to study state 6 hours.




GRAPH 4 - Dilatometric study of 1st and 2nd stage graphitization of test specimen from Group 9

Temperature 1700 F

Time to study state 3 hours.




**GRAPH 5** - **Annealing Cycle**

The annealing was carried out in a vacuum of 30 to 50 microns for two reasons. The first in order to eliminate if possible any oxidization of the specimens that might cause surface defects, and hence instigate error in the tensile test. The second because it has been reported, (28) the most

(28) Controlled Atmosphere Annealing of Malleable Iron, Report of the AFS Malleable Div. Sub-Committee, AFS Preprint, No. 45-49, 1949.

rapid annealing of malleable iron takes place in vacuum.

The annealing furnace was one designed and constructed at Missouri School of Mines. Temperature was controlled by a Wheelco Capacitrol and calibrated -- thermo couples. The vacuum was maintained by a Kinney Hi Vacuum oil pump in series with a vacuum diffusion pump made by the National Research Corporation. The relative vacuum was measured by a Thermocouple Gage Control made by the Vacuum Engr. Div. of National Research Corporation.

Tensile Tests: The tensile tests were conducted on two machines: a Tinius Olsen Universal Hydraulic Testing machine, using the 30,000 lb. range, and a Southwark-Tate-Emery 20,000 lb. capacity hydraulic testing machine. Before use, each machine was calibrated with a Morehouse elastic proving ring.

As recommended in ASTM standard A 47-47 a stress producing an elongation under load of 0.01 inches over the gage length of 2 inches was considered the yield point.

To measure this elongation, the Moore Extensometer (a

dial type strain gage using a 2 inch gage length with a 5-1 magnification) was used. It is capable of recording strain in the order of 0.0002 inches per 2 inch gage length. On several specimens (6b, 8c, 12b) a stress-strain curve was recorded using a Templin type - O.S. Peters electrical extensometer, and a Templin recorder Type TA-1. (See graphs No. 7, 8, 9, pages 30, 31 and 32.)

Division of Bars: One representative test bar from each test group was sectioned in order to obtain specimens for metallographic examination, charpy impact and coercive force tests.

Charpy Impact: A Tinius Olsen Impact testing machine was used. Charpy impact specimens were machined from the 3/4 inch diameter grip ends of the tensile test bars. The specimens were machined following the ASTM standard E 23-47T, section 9. Simple beam impact specimens were used, employing the "keyhole" notch.

Coercive Force: Coercive force specimens were machined from the 5/8 inch section of the test bar, as close as possible to the break. Coercive force readings were taken on a Model 2 Coercimeter as described by V. H. Gottschalk. (29)

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(29) Gottschalk, V.H., Development and Application of the Coercimeter, Bureau of Mines Report of Investigation, # 3400.

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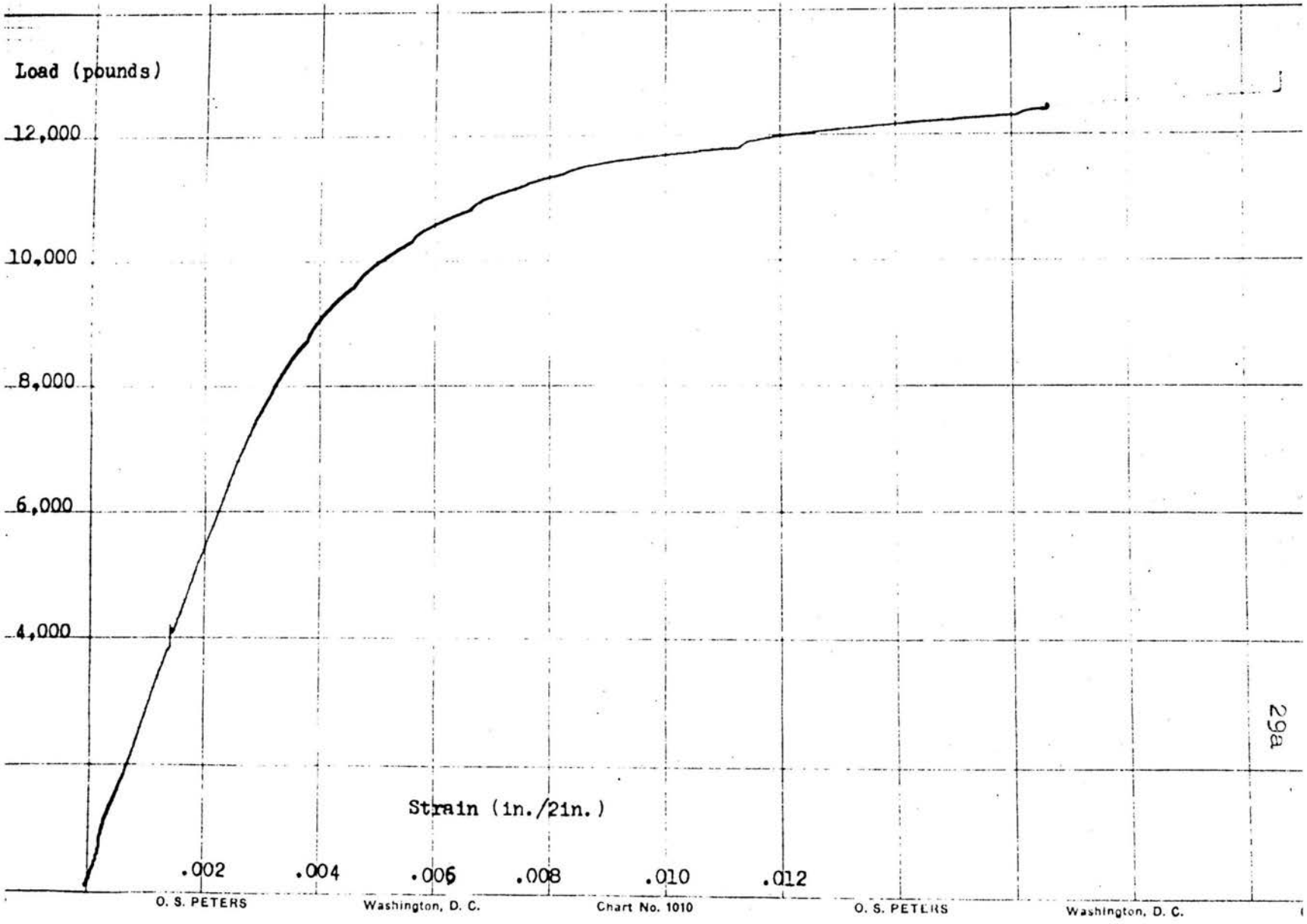
Brinell Hardness: The Brinell hardness readings were taken on a standard Tinius Olsen machine.

Micro-hardness: Micro-hardness readings were taken using

GRAPH 6

Load-Strain curve for specimen No. 12b

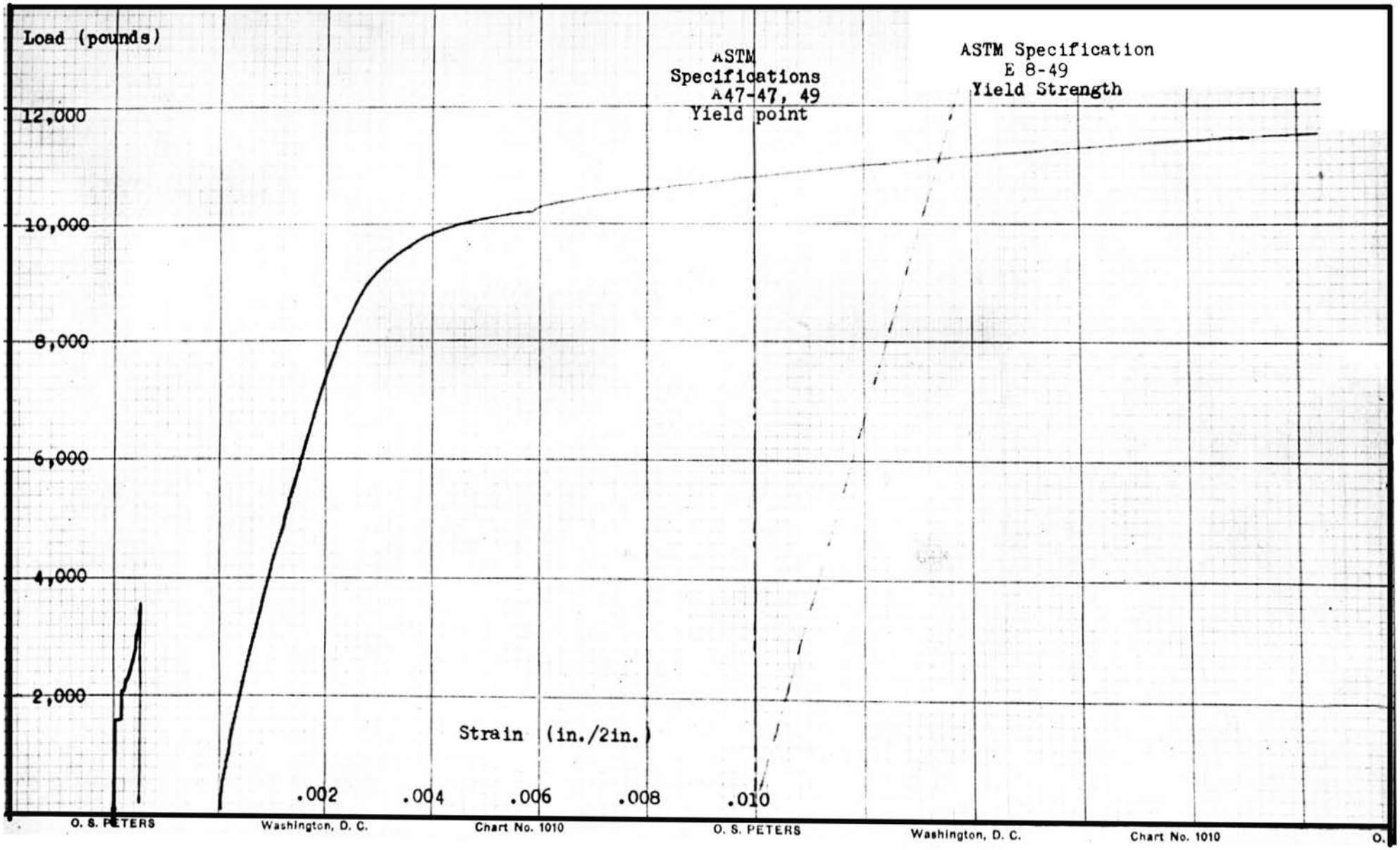
Plotted by Templin recorder



GRAPH 7

Load-Strain Curve for specimen No. 6b

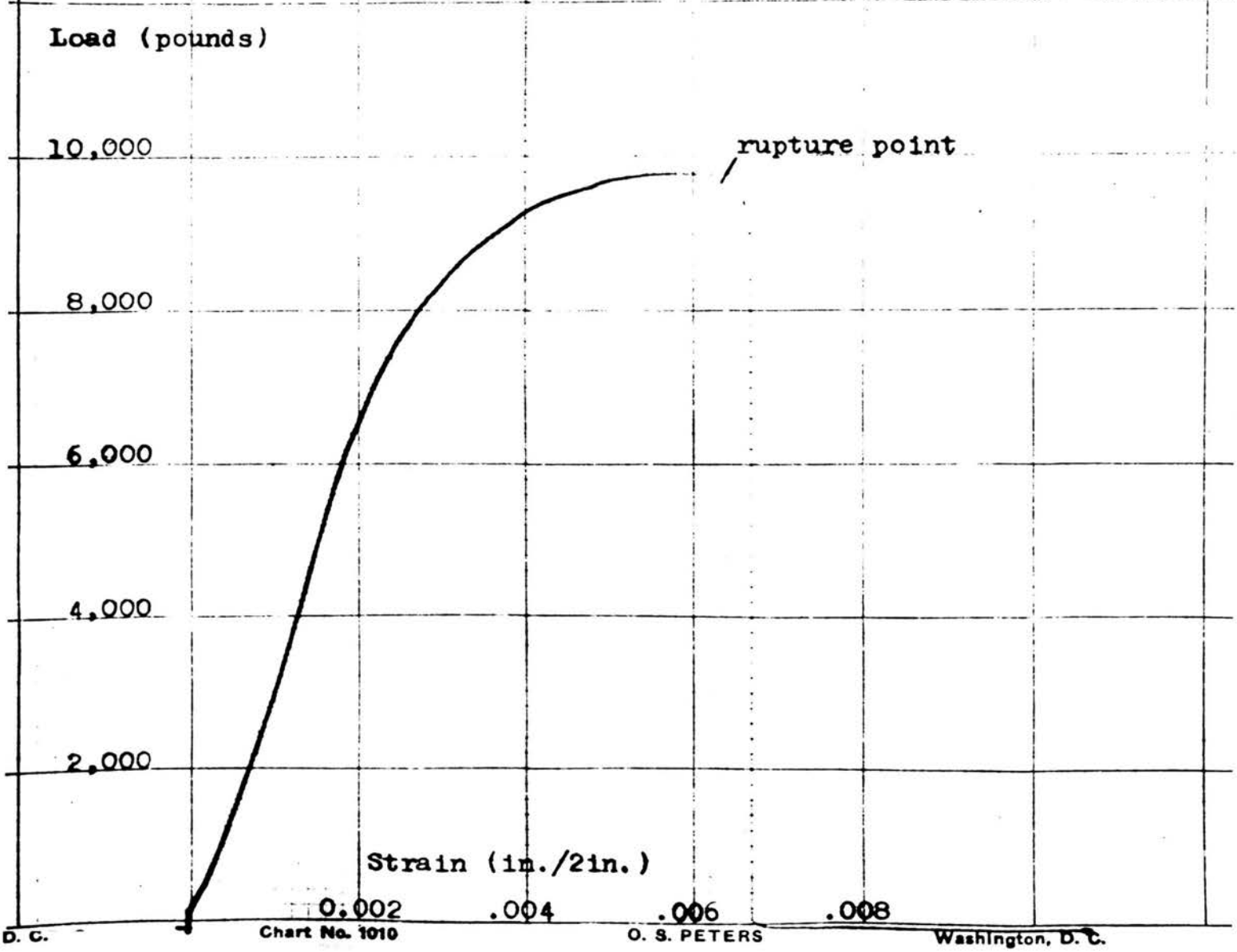
Plotted by Templin recorder



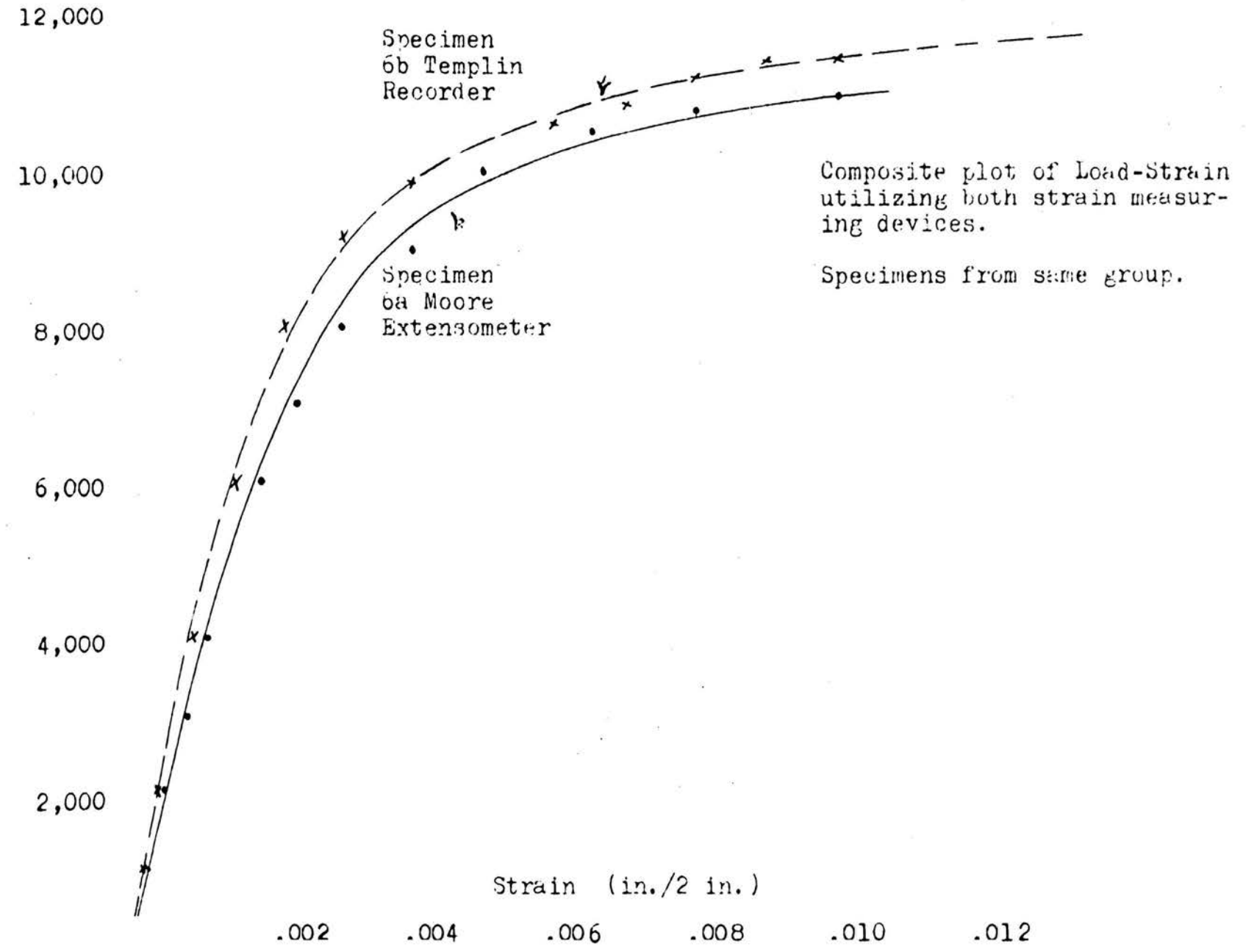
GRAPH 8

Load-Strain Curve for specimen No. 8c

Plotted by Templin recorder



GRAPH 9


Load (pounds)
12,000
10,000
8,000
6,000
4,000
2,000
Specimen
6b Templin
Recorder
Specimen
6a Moore
Extensometer
Composite plot of Load-Strain utilizing both strain measuring devices.
Specimens from same group.
Strain (in./2 in.)
.002
.004
.006
.008
.010
.012

Model LR Tukon Hardness Tester with a Vickers diamond indenter.

Metallographic specimens: The metallographic specimens were cut as close as possible to the face of the break. One bar was also sectioned lengthwise. A flat surface was prepared on an emery wheel, and polishing was accomplished with standard papers and diamond dust. (See Appendix for Methods).

Nodule Count: The nodule counts were made at 50X, and are the average of four areas all within 1/8 inch of center of each bar. This was done in order to eliminate the error in counting areas that may have oxidized, hence contain less carbon.

Chemical Analysis: The chemical analysis for combined and free carbon were performed by the Frank L. Crobaugh Company laboratories.

# EXPERIMENTAL DATA

A summation of the results from all experimental tests will be found in Master Table B, page 35.

| Group | Specimen | Pretreat Hardness (Rc) | Charpy Impact (ft lb) | R/A % ave. | Yield Strength (psi) | Tensile Strength (psi) | % Elong. 2 in. | Tukon Hardness (Vickers) Stressed | Tukon Hardness (Vickers) Relieved | Mean Tensile Strength | Ferrite gr. size (Microns) | Estimated pearlite (%) | Combined Carbon (%) | Free Carbon (%) | Nodule No. per sq. mm | Coercive Force (5/8 sect.) (oersteds) Stressed | Coercive Force (5/8 sect.) (oersteds) Relieved | Coercive Force (3/4 sect.) (oersteds) Stressed | Coercive Force (3/4 sect.) (oersteds) Relieved | Group and Specimen |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| I | a | 38-40 | 10.8 | 12.1 | 40,300 | 57,400 | 9.0 | 145 | 129 | 56,300 | 20-40 | 5-10 | 0.12 | 2.39 | 90 | 3.90 | 2.32 | 3.62 | 3.4 | Ia |
| | b | | | | 39,900 | 55,400 | 11.5 | | | | | | | | | | | | | b |
| | c | | | | 40,700 | 56,000 | 11.5 | | | | | | | | | | | | | c |
| II | a | 32-35 | 14.0 | 15.5 | 39,900 | 56,800 | 12.0 | 148 | 127 | 57,000 | 40-60 | 5 | 0.10 | 2.40 | 120 | 3.62 | 2.04 | 2.94 | 2.83 | IIa |
| | b | | | | 40,100 | 57,800 | 11.0 | | | | | | | | | | | | | b |
| | c | | | | 40,100 | 57,400 | 12.5 | | | | | | | | | | | | | c |
| III | a | 32-36 | 6.0 | 11.5 | 43,000 | 61,700 | 10.0 | 151 | 110 | 60,600 | 40-60 | 30-40 | 0.57 | 2.00 | 75 | 4.92 | 2.38 | 4.29 | 4.07 | IIIa |
| | b | | | | 42,000 | 61,000 | 9.8 | | | | | | | | | | | | | b |
| | c | | | | 41,000 | 59,000 | 10.0 | | | | | | | | | | | | | c |
| IV | a | 34-37 | 11.0 | 17.0 | 38,050 | 55,800 | 10.5 | 143 | 125 | 58,000 | 20-40 | 15 | 0.05 | 2.49 | 90 | 3.96 | 1.81 | 4.29 | 4.07 | IVa |
| | b | | | | 41,000 | 59,400 | 8.0 | | | | | | | | | | | | | b |
| | c | | | | 39,400 | 57,700 | 10.0 | | | | | | | | | | | | | c |
| V | a | 35-38 | 8.0 | 15.8 | 43,200 | 63,700 | 9.2 | 139 | 110 | 62,000 | 20-40 | 30-40 | 0.90 | 1.56 | 140 | 4.47 | 2.94 | | 4.41 | Va |
| | b | | | | 40,500 | 60,300 | 8.6 | | | | | | | | | | | | | b |
| | c | | | | 42,500 | 63,00 | 10.0 | | | | | | | | | | | | | c |
| VI | a | 38-41 | 7.8 | 12.0 | 38,200 | 56,500 | 13.5 | 143 | 126 | 55,800 | 40-60 | 0 | 0.06 | 2.53 | 100 | 3.40 | 1.53 | | | VIa |
| | b | | (5.8) | | 40,100 | 55,100 | 14.0 | | | | | | | | | | | | | b |
| | c | | | | 37,700 | 55,600 | 15.0 | | | | | | | | | | | | | c |
| VII | a | 50-55 | 8.0 | 5.0 | 40,100 | 62,400 | 5.0 | 147 | 126 | 58,000 | 10-15 | 0 | 0.04 | 2.60 | 4,000 | 5.57 | 2.94 | 3.62 | 2.05 | VIIa |
| | b | | | | 39,600 | 55,200 | 4.0 | 147 | | | | | | | | | | | | b |
| | c | | | | - | - | - | | | | | | | | | | | | | c |
| VIII | a | 62-67 | 7.2 | 3.7 | 35,200 | 37,200 | 0 | 148 | 130 | 59,200 | 10-15 | 0 | 0.04 | 2.54 | 8,000 | 3.62 | 3.16 | 2.94 | 2.89 | VIIIa |
| | b | | | | - | 59,200 | 0.5 | | | | | | | | | | | | | b |
| | c | | | | - | 35,000 | 0 | 133 | | | | | | | | | | | | c |
| IX | a | 65-67 | 10.0 | 4.0 | - | 63,350 | 0 | 128 | 113 | 62,250 | 8-12 | 0 | 0.04 | 2.48 | 10,000 plus | 5.77 | 3.17 | 4.29 | 2.94 | IXa |
| | b | | | | 37,200 | 38,000 | 0 | 148 | | | | | | | | | | | | b |
| | c | | | | - | 62,200 | 0 | 130 | | | | | | | | | | | | c |
| X | a | 42-44 | 4.0 | 11.5 | 37,400 | 54,200 | 9.5 | 147 | 101 | 55,000 | 40-60 | 5 | 0.55 | 1.89 | 180 | 3.74 | 2.21 | 3.25 | 3.12 | Xa |
| | b | | | | 36,500 | 53,400 | 9.2 | | | | | | | | | | | | | b |
| | c | | | | 38,100 | 56,500 | 11.5 | | | | | | | | | | | | | c |
| XI | a | 40-44 | 9.2 | 11.7 | 38,600 | 56,000 | 10.0 | 154 | 108 | 57,500 | 20-40 | 5 | 0.45 | 2.17 | 135 | 4.18 | 2.26 | 3.62 | 3.51 | XIa |
| | b | | | | 41,700 | 59,000 | 10.5 | | | | | | | | | | | | | b |
| | c | | | | 39,800 | 57,500 | 11.5 | | | | | | | | | | | | | c |
| XII | a | 37-40 | 5.0 | 11.7 | 40,000 | 59,500 | 10.0 | 135 | 105 | 60,000 | 20-40 | 30-40 | 0.07 | 2.54 | 75 | 4.98 | 2.38 | 4.18 | 4.18 | XIIa |
| | b | | | | 42,500 | 60,900 | 10.0 | | | | | | | | | | | | | b |
| | c | | | | - | 61,000 | 7.8 | | | | | | | | | | | | | c |
| XIII | a | 36-40 | 5.5 | 12.0 | 37,900 | 54,700 | 11.5 | | | 55,500 | 40-60 | 30-40 | 0.09 | 2.39 | 80 | 4.35 | 2.32 | 3.73 | 3.45 | XIIIa |
| | b | | | | 39,050 | 55,200 | 11.0 | | | | | | | | | | | | | b |
| | c | | | | 39,200 | 56,400 | 10.5 | | | | | | | | | | | | | c |
| XIV | a | 41-43 | (15.5) | 7.5 | 36,600 | 52,000 | 6.5 | 158 | 144 | 52,600 | 10-15 | 0 | 0.04 | 2.54 | 250 | | | | | XIVa |
| | b | | | | 37,400 | 52,600 | 4.0 | | | | | | | | | 2.72 | 1.3 | 1.80 | 1.13 | b |
| | c | | | | 38,000 | 54,400 | 8.5 | | | | | | | | | | | | | c |

## DISCUSSION OF RESULTS

Before considering the specific results for each group, some general explanation of several of the tests is worthwhile.

Thermal pre-treatment: The thermal treatments which comprise groups 1 to 14 (see Graph 4), were selected to investigate specific observations reported in the literature.

Group 1, in the "as cast" condition, is considered as unity, or a basis for the other pre-treatments.

Group 2, the 800° F relief treatment, was selected to illustrate the phenomenon reported by Palmer, (30) Hall, (31) and Hultgren and Edstrom (32). Slow heating through, or

(30) Palmer, S.W., op. cit., p. 24.

(31) Hall, H.G., Malleable Cast Iron, Proc. Inst. of British Foundrymen, 1946-47.

(32) Hultgren, A. and Edstrom, O., op. cit., p. 15.

pre-treatment within the temperature range of 700 to 1050 F will increase the number of temper carbon nodules resulting from the annealing. This is an unique method, for all other pre-treatments add energy to the system (cold work, quenching, alloy additions) while this method is believed to relieve the solidification stresses set up in the casting on freezing. The British Cast Iron Research Association is now

working in this field, but as yet have published no data concerning it.

Groups 3, 4, and 5 were included to observe the effect of air quenching on nodule count. Scott (33) has investigated

(33) Scott, Howard, Quenching Media, Trans. ASM 22, p. 577, 1934.

the use of air as a quenching medium, and reports cooling rates with reference to water at 65 F from .018 to 0.7 at 1330 F. This method is perhaps important in that air stream quenching could readily be applied to commercial foundry practice.

Groups 6, 7, 8, and 9 were used because it is possible, using water at a known temperature, to ascertain the quenching rate, or cooling power of the solutions relative to that of water at 65 F. (The cooling rate of water at 65 F is considered as unity, and is 3260 F per second through the range from 1330 to 1020 F.) Using water at 65 F as unity, the relative cooling rate of Gp. 6 (water at 212 F) is 0.044, Gp. 7 (water at 120 F) is 0.17, Gp. 8 unity, Gp. 9 (10% NaCl solution at 65 F) 2.0.

Groups 10, 11, 12 and 13 demonstrated the effect of variation of quenching temperature upon the subsequent nodule count. The quenching medium used was water at 212 F.

Group 14, the oil quench was included to note any possible difference in quenching media upon the physical properties of the resulting malleable iron. Hultgren and Edstrom (34)

(34) Hultgren, A., and Edstrom, O., op. cit., p. 15.

studied the effect of pre-quench treatments of white iron in different media, using water, oil, liquid air, molten lead-tin, and molten lead. They concluded the presence of some martensite in the pre-quenched structure was a necessary condition for the pre-quench treatment to have any effect.

If the relative hardness of the pre-treated bars used in this investigation, is indicative of the amount of martensite present, it is noted that the hardness of the "as cast" bars was 38-40 Rc. Pre-treatment resulted in a hardness range from 32 to 67 Rc, with gaps at 44-50 and 55-62. Simmons (35)

(35) Simmons, O.W., Quenching Rate Versus Graphite Formation in Prequenched White Cast Iron, Trans. ASM 32, p. 255, 1944.

performed a unique experiment in 1943, a white cast iron cylinder heated to 1635 F, was subjected to a quench with a stream of water at 75 F directed against a ground end in a manner following the procedure employed for the end-quench hardenability test. The cylinder was then sectioned longitudinally, hardness readings taken, and the flat section prepared for metallographic examination. It was found that -- a Rockwell hardness of 57 Rc or more attained by a cooling rate of more than 240 F pwer second at 1300 F was necessary to give martensite which annealed into a structure having a very fine prequenced type of graphite. In a comparatively narrow cooling range of 170 to 130 F per second

at 1300 F, Rockwell C hardness values of 56 to 54 respectively were obtained. The martensite formed in this range is interspersed with a constituent appearing to be fine pearlite and the resulting graphite changes from the very fine pre-quenched type, to a variety that closely resembles that found in ordinary malleable iron, though somewhat finer and more numerous. As slower cooling rates are reached, the increase in the graphite size is gradual.

The hardness of the white iron in Simmons investigation was 44 Rc. From his work it may be deduced that a hardness increase of 13 points Rc is necessary for very fine nodules. In the groups considered in this investigation Numbers 7, 8, and 9 have this hardness increment. If Table B is consulted it can be seen that these groups have very fine nodules. To cause a definite increase in nodule count, a hardness increase of 10 points Rc is needed, numbers 6, 10, 11 and 14 would fall into this class. If Table B is again consulted it will be seen that these groups do have an increased nodule count.

Original and Final Diameters: The measurement of the diameters of the bars present the usual difficulties. They were in the "as cast" condition, and due to commercial casting techniques none were perfectly cylindrical. The variations in diameters were as great as 0.020 inches. In each case an attempt was made to select a mean diameter, but it is believed that the accuracy is no greater than plus or minus 0.005 inches in any bar. This will cause a possibility of

error in the yield point, and tensile strengths of plus or minus 1000 psi.

Reduction of area: The reduction of area of "as cast" bars has little meaning in that if the bars are out of round before the tensile test, after the test their form is ellipsoidal.

The American Malleable Iron Handbook indicates that malleable iron does not tend to "neck" in breaking, but rather stretch along the entire gage length. This statement has been confirmed in this investigation. Reduction of Area is given as a relative value.

Yield Point: The (ASTM) Standard Specification for Malleable Iron Castings, A47-48. States in section 4b; "The yield point may be determined by the drop of the beam or halt in the gage of the testing machine, or by dividers method. If determined by the dividers method, the stress producing an elongation under load of 0.01 inch over the gage length of 2 inches shall be taken as the yield point value.

There is no "yield point" in malleable iron if the ASTM definition of yield point is considered, i.e., there is no marked increase in strain without an increase in stress. In conducting the tensile tests for this investigation, no test bar exhibited a yield point. See (Graphs 6, 7, and 8, pages 29a, 30, 31) and also (Graph 9, page 32.) In Graph 9 the solid line marked (ASTM 47-47) indicates the value actually used in each test to obtain the yield point. Note it is not the true yield value. The closest definition to

this value is possible "yield strength", the stress at which a material exhibits a specified limiting permanent set, (dashed line marked ASTM E8-49). However, this also would not be strictly true in that the method used in the tests shows an elongation under load, with no reference to permanent set.

R. D. Landon [36] reports the "Yield Point" might better

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(36) Landon, R.D., Stress-Strain Relations for Malleable Cast Iron in Tension with Special Attention to Yield Point Determinations, Proc., ASTM, Vol. 40, p. 849, 1940.

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be expressed as a percentage of the ultimate stress value, rather than a fixed value. He also proposes "Proof Stress" be used for the stress to be equalled or exceeded at a definite, measured elongation. His work also considers the fact that there was no drop of the beam of the testing machine, or dip in the stress strain diagram.

Graph (9): Graph 9 (page 32) is presented to indicate the close symmetry of load strain curves, one produced by the Templin Recorder, and the other with values given by the Moore Extensometer.

Tukon Hardness: Where ever possible the hardness values taken were for single grains. If the grain size was very fine, an attempt was made to project the point of the Vickers diamond indentor in the center of a grain, and not in a grain boundary. The values presented in Table B are average values for each specimen tested. Depending on the selection of grains, and the position over the cross section of the specimen, it

was possible to obtain a hardness variation of 30 points on the Vickers scale. However, by careful selection of areas, the hardness values could be held to a plus or minus 5 points per specimen. Grain orientation is believed to be the cause of this variation. Specimens 6b, and 12a will serve to illustrate the extremes in that the average Vickers hardness for (6b) is 140, but a reading of 77 Vickers could be found if large single grains were selected. Specimen (12a) also exhibited this in that the average hardness was again 130 to 140 Vickers, but readings of 73.3 Vickers hardness were recorded.

The hardness values for specimens 7, 8, and 9 are a compromise in that they represent the hardness of the ferrite and graphite combined. The grains were so small, and the graphite finely dispersed, that no area could be found where the hardness of one constituent could be taken separately. It is felt that the hardness of the ferrite was much greater than is actually represented.

Tensile Strength: Specimens number 5a, 7c, and 8b broke at the grip ends, however it was possible to re-grip No. 5a and pull it to rupture. Micro-examination of the polished fracture at the grip end of 7c indicated a severe dendritic growth projecting from the casting gate into the center of the bar. Specimen No. 8b broke at the grip end under 59, 200 psi, no defect in the fracture.

Specimens number 7a, 7b, 9c, 14a and 14b revealed a peculiar fracture, in that the over-all fracture was grey, but

at one side starting at the outer edge of the fracture a dark circle was generated. This could possibly indicate a surface defect, non-parallel gripping of the bar, or segregation of the base metal.

Specimens number 8a, 8c, and 9b exhibited what is believed to be a multi-stress fracture due to the water quenching. Alternate dark and light rings radiated from the center of the fracture. The center was light grey to silvery, next came a dark ring, followed by another light grey area continued to the outer edge of the fracture.

It is known, (37) water quenched steels are under high

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(37) The ASM Metals Handbook, p. 616.

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compressive stress at the surface, and tension in the core, chiefly because the density of martensite is less than that of pearlite. Such stresses, however, are not dangerous and may even be beneficial <u>in a steel</u> if the core is sound and if no holes bring a surface into the tension zone. (Cracks occur under tensional stress only.) Since cracks occur under tensional stress only, and malleable iron has many temper carbon nodules, (holes) throughout the cross section, it is believe severe water quenching may cause some minute cracks either during the quench, or in the first stage of the annealing cycle before the bar has undergone complete recovery from the stressed condition. If the specimen were subjected to tension at a slow rate these minute cracks would have time to grow and weaken the piece considerably. Also during

this slow rate of tension, work hardening due to the temper carbon nodule, (hole) acting as a stress concentrator could effect the piece more than at a faster rate. If groups 8 and 9 are considered this may be substantiated by the fact that specimens 8a, 8c and 9b were pulled at a very slow rate with strain gages attached. (See Graph No. 8, page 31). Specimen number 8c broke shortly after the yield strength was reached as verified by the Templin recorded plot of this specimen. Specimen number 9b also broke as the yield strength was reached. These specimens could withstand the elastic deformation, however when in the plastic range, the overall hardness, due to work hardening, presented a brittle material, through which the quenching cracks could extend more easily than they could through a ductile material hence rupture occurred prematurely.

It should be noted here that group 14 specimens also ruptured prematurely, however in this case it is believed the loss of strength was due to a dendritic grouping of the temper carbon nodules forming effectively quasi dendritic stringers of graphite. The stresses that develop in oil quenching are lower than the residual stresses resulting from water, but dangerous because the residual stresses in oil quenched material are usually tensional at the surface, and compressive at the core. Specimens 14a and 14b fractures have possible defects radiating from the surface.

Non-Homogeneity of Certain Test Bars: It appeared that segregation of the basic white cast iron during solidification may

have taken place in several test bars.

Specimen 14c broke at a tensile strength of 54,500 psi with an elongation of 8.5%. Micro-examination of the polished rupture showed fine nodules, grouped in a dendritic pattern, with a matrix of ferrite. Chemical analysis of a coercive force specimen taken within 0.5 inches of the break indicated 0.04% combined carbon. An impact specimen, taken from one of the 3/4 inch grip ends, broke at 15.5 ft. lbs., too high a value for malleable iron. A polished and etched surface of the impact specimen indicated a microstructure of white iron (pearlite and cementite). The other 3/4 inch grip end of test bar 14c was then polished and etched, and exhibited a normal malleable structure similar to the photo-micrograph near the point of rupture.

Specimen 5b was reported as having 0.9% combined carbon, equivalent to approximately 100% pearlite plus 2.5% cementite. Metallographic examination of a sample taken within 0.5 inches of this spot indicated at the maximum 30 to 40% pearlite, and no cementite.

Coercive Force: Coercive force is the strength of a magnetic field required to reduce the magnetization of a specimen to zero. Graphically it is the negative intercept along the (x) axis of a hysteresis curve. Physically it is the energy required to cause complete de-magnetization of a crystal lattice. Since it is a function of the type of atom, and the atomic arrangement, then lattice strain, and non-metallic inclusions should be factors that would affect the actual

coercive force values.

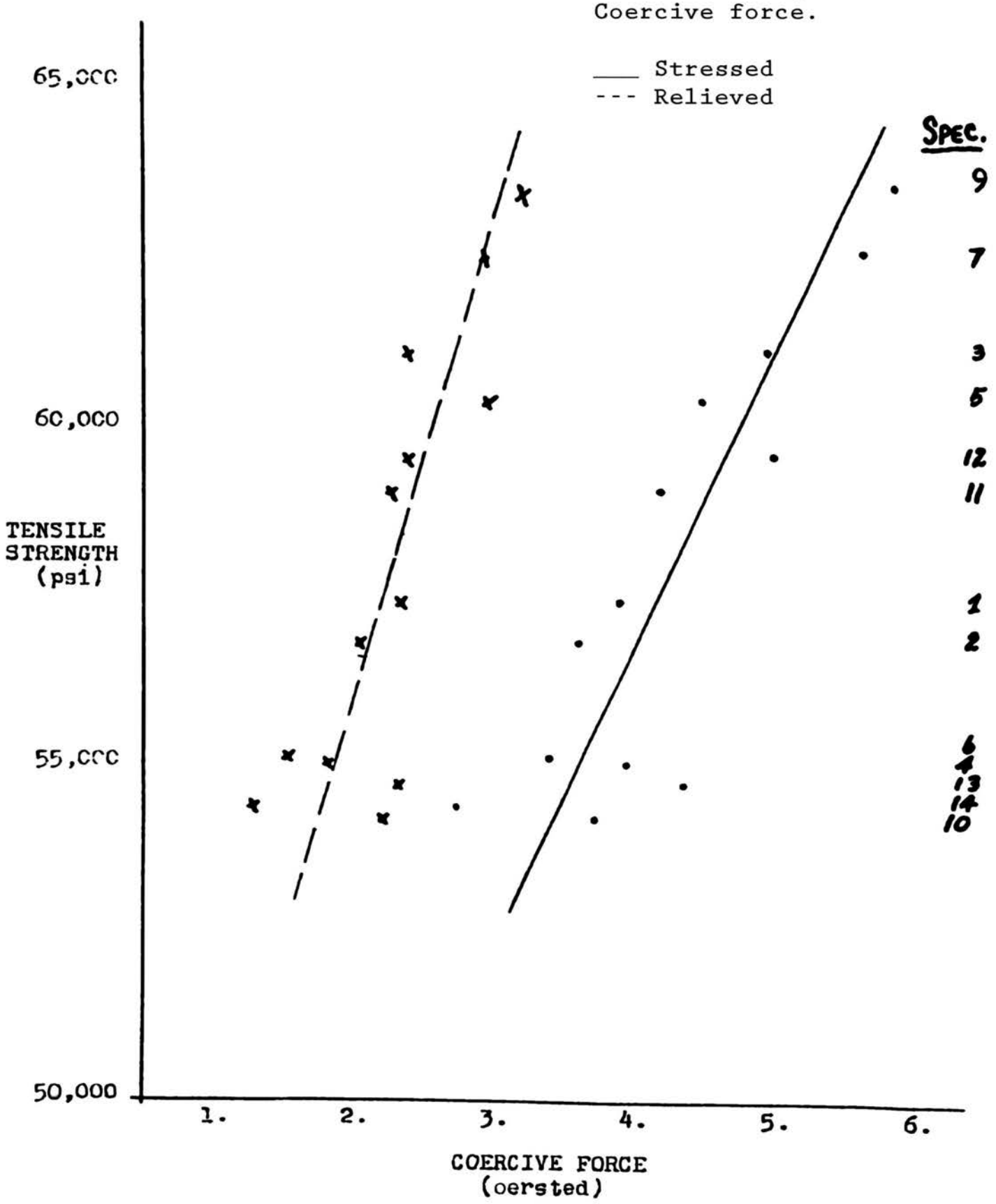
Dr. Epplesheimer suggested this property could possibly be used to measure the nodule size and distribution, and the internal stress in each test bar. In order to test this possible relationship, two separate test specimens were cut from a representative test bar from each group. One specimen was taken near the fracture in the 5/8 inch section, and the other from the 3/4 inch grip ends.

Coercive force values were obtained from each set in the stressed (or as pulled) condition, then each set was relieved at 1200 F for four hours and allowed to cool to room temperature in 12 additional hours. Coercive force readings were again obtained. These readings are tabulated in Table B. The listing includes both the stressed and relieved values for both the 5/8 inch and 3/4 inch sections. This data is also presented graphically (Graphs 10, 11, 12, and 13, pages 47, 48, 49 and 50).

Graph No. 10: Graph No. 10 presents the relationship between tensile strength and coercive force. Specimens taken from the 5/8 inch section of the tensile test bars, as near as possible to the break. The plot with the higher coercive force readings (solid line and dots) represents the highly stressed condition of the bars as pulled. The plot to the left, hence lower coercive force readings are the same specimens after being subjected to a stress relieving treatment.

By comparing the coercive force readings of the stressed,

Graph 10
Tensile strength -vs-
Coercive force.

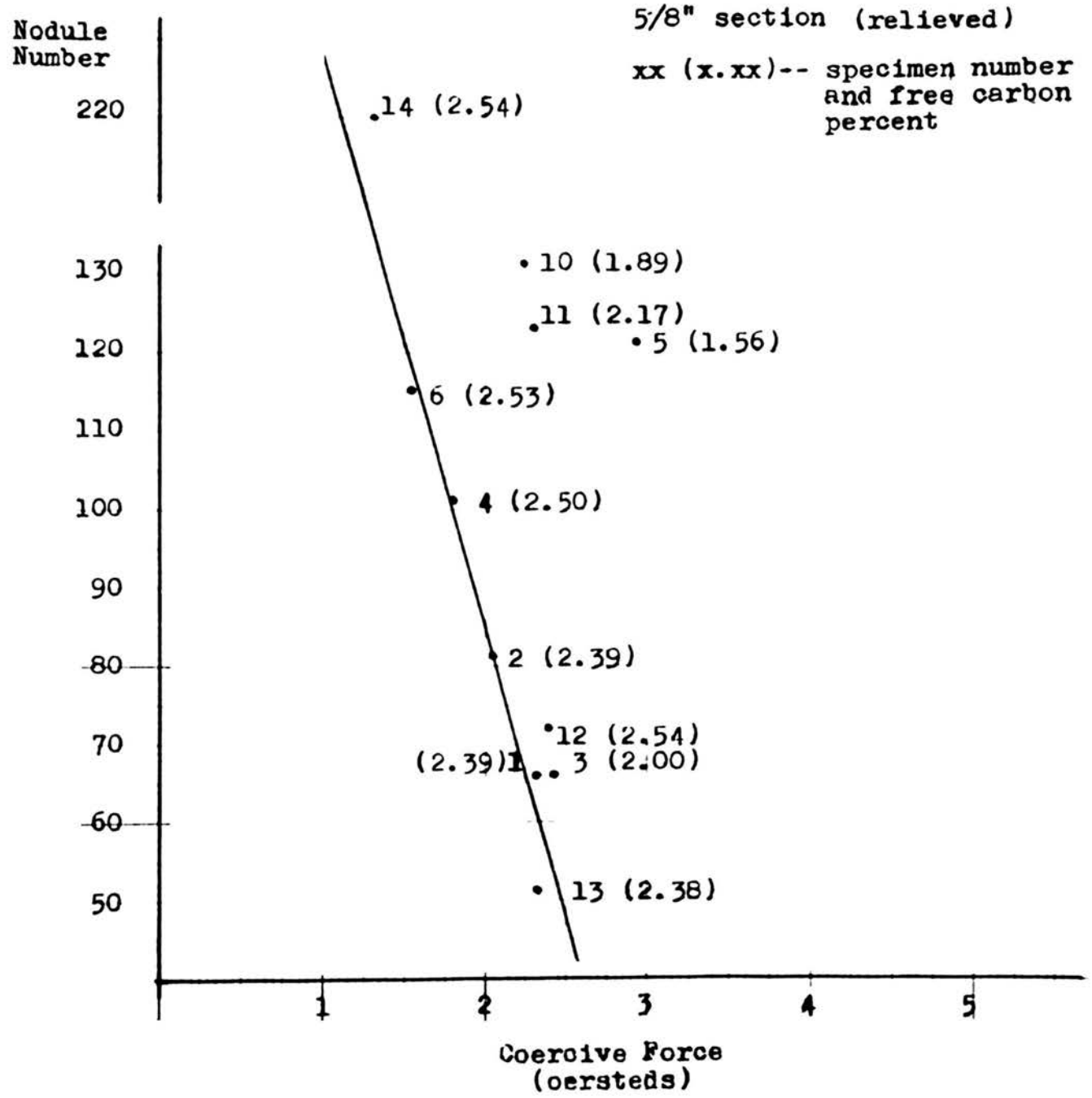


**GRAPH 11**

Nodule count -vs- Coercive Force

5/8" section (relieved)

xx (x.xx)-- specimen number and free carbon percent

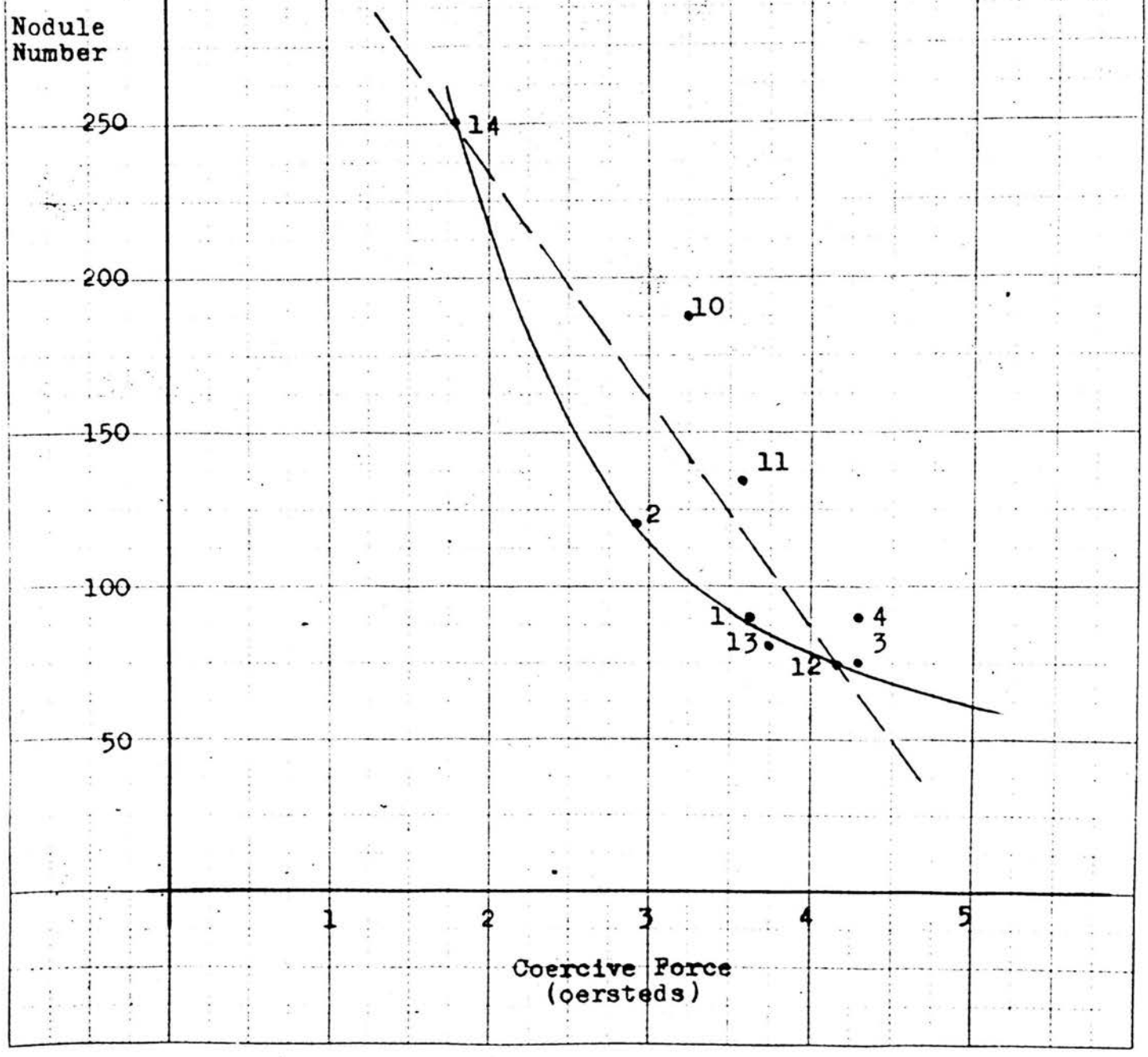


**GRAPH 12**

Nodule count -vs- Coercive Force

3/4" section (as pulled)

xx specimen number

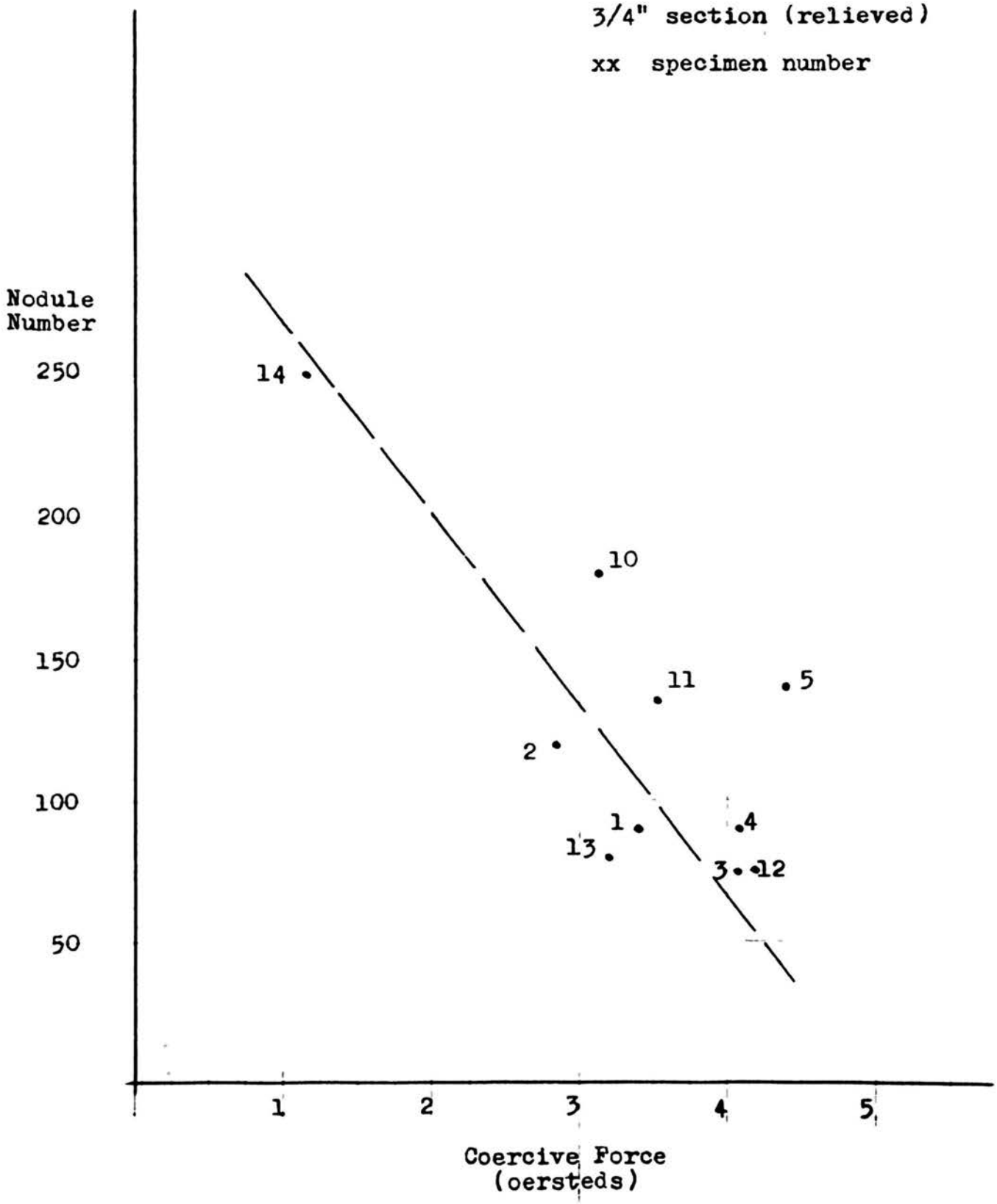


GRAPH 13

Nodule count -vs- Coercive Force

3/4" section (relieved)

xx specimen number



and relieved specimens it is possible to ascertain if the original test bar ruptured at a maximum stress, or if a defect in the bar caused premature rupture at a lower stress. It should be noted that the relief treatment affected the highly stressed bars to a greater extent than the bars that broke at a lower stress concentration (tensile strength). However, the effect is proportional to the stressed condition.

If Table B is consulted for the chemical analysis of the coercive force specimens, it is noted, Specimens 3, 5, 10, and 11 contain considerable amounts of combined carbon, and the free carbon varies from 1.89 to 2.54%. This should indicate that the matrix structure and the nodule size or number do not effect the relationship in the stressed or relieved condition.

It is felt that the correlation between tensile strength and coercive force, for the malleable iron test bars used in this experiment, is of a high order.

Graph No. 11: The stress relieved specimens of Graph 10 were polished, and subsequent nodule counts taken of the cross section. (In each specimen the nodule count was taken at the center). Graph 11 presents the relationship between Temper carbon, Nodule Number versus Coercive force values.

From this plot it may be seen that there appeared to exist a definite relationship between the nodule count, and coercive force. Chemical analysis of the coercive force specimens for free and combined carbon reveals Specimens 10, 11, and 5 (that appear to be out of place) each have low free car-

bon, 1.89%, 2.17%, and 1.56% respectively. It is felt that this lower free carbon content definitely effects this relationship. Hence these three values were disregarded.

Graph No. 12 and 13: Graphs No. 12 and 13 were attempts to relate the temper carbon nodule number with coercive force. The coercive force specimens for these graphs were taken from one of the 3/4 inch grip ends of the same test bars as plotted in graphs 10 and 11.

Graph 12 is a plot of the temper carbon nodule number versus the coercive force of specimens, from the 3/4 inch grip end sections, in the "as pulled" condition. These specimens should not have been subjected to any stress in the tensile machine.

With the exception of specimens 10, 11, and 4 a correlation again was noted, however in this case the relationship was of a higher degree, indicating a third variable might be involved. This could have possibly been either residual stress, varying free carbon, or segregation of the matrix structure. If an arbitrary straight line is drawn through the plotted values in an attempt to produce a mean function, it is found: the slope of this line (dash line on graph) is such that 1 oersted (coercive force) equals 75 nodules. This closely follows the original relieved relationship (Graph 11) which has a slope of 1 oersted equals 70 nodules.

Graph 13, represents the same specimens as Graph 12, but in this case the specimens were subjected to a stress relief treatment in an attempt to simplify the curve, and possibly

eliminate the third function. The results were inconclusive, and tended to complicate the relationship further. An arbitrary straight line drawn through the points gave a slope of 1 oersted equals 65 nodules, however this has little meaning in that the points were too far apart to show any positive results.

The ends of each of these coercive force specimens were prepared for metallographic examination, and found to contain varying amounts of pearlite, and possibly cementite. It is believed that matrix structure and segregation cast doubt on the results obtained in Graph 13.

It is believed that there is a correlation between temper carbon nodule number and coercive force. This relationship is definitely dependent upon the matrix structure, and free carbon. Graph 11 and Graph 12 are believed to support this relationship. More work should be done in order to verify the results as presented in this investigation.

Photomicrographs from each of the groups will be found of pages 54 to 68. They are listed by groups, Fig. (1a) and (1b) are from group 1, Fig. (2a) and (2b) are group 2, etc. The (a) photomicrographs were sections taken from the 5/8 inch coercive force specimens magnification 50X, unetched, The (b) photomicrographs were taken from the test bars, at the rupture, parallel to the break. Magnification 250X, etched 8 seconds in 4 % nital.

The dull grey inclusions, as seen in each of the etched plates, have been identified as a manganese sulfide compound.

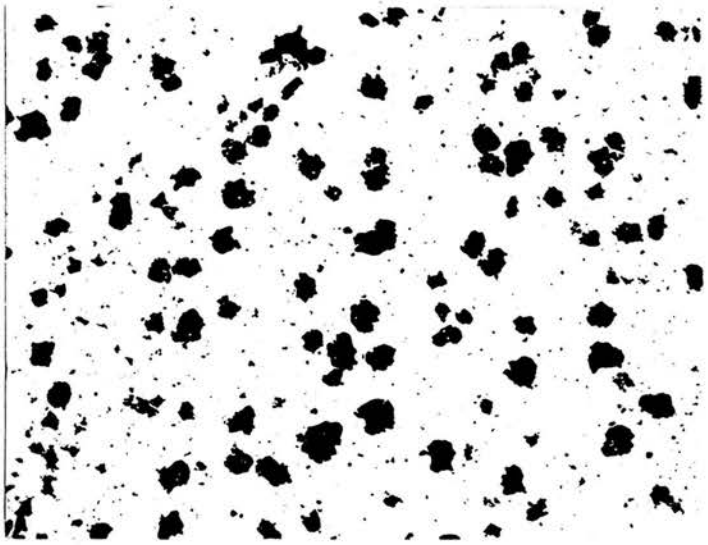


Fig. 1a Mag. 50X

Group 1 Specimen a

Unetched

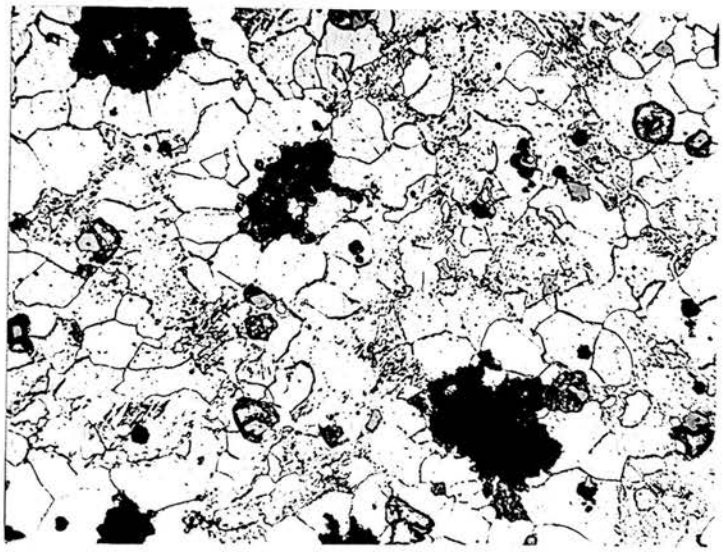
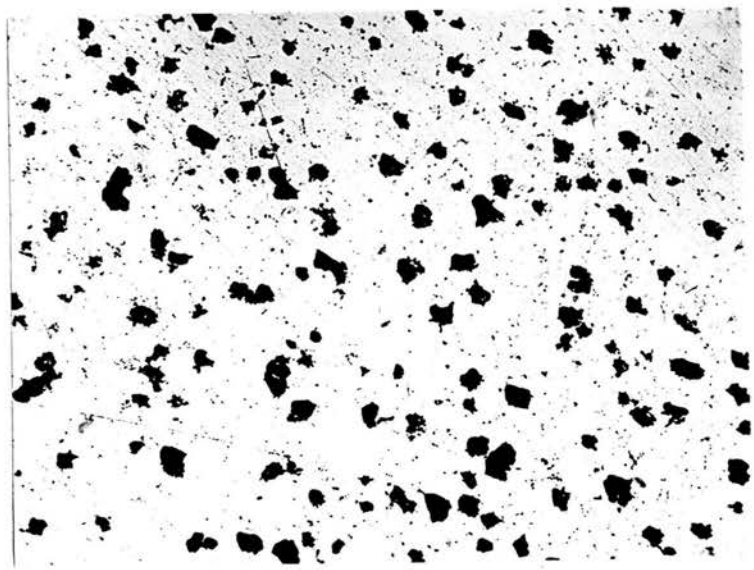


Fig. 1b Mag. 250X

Group 1 specimen a

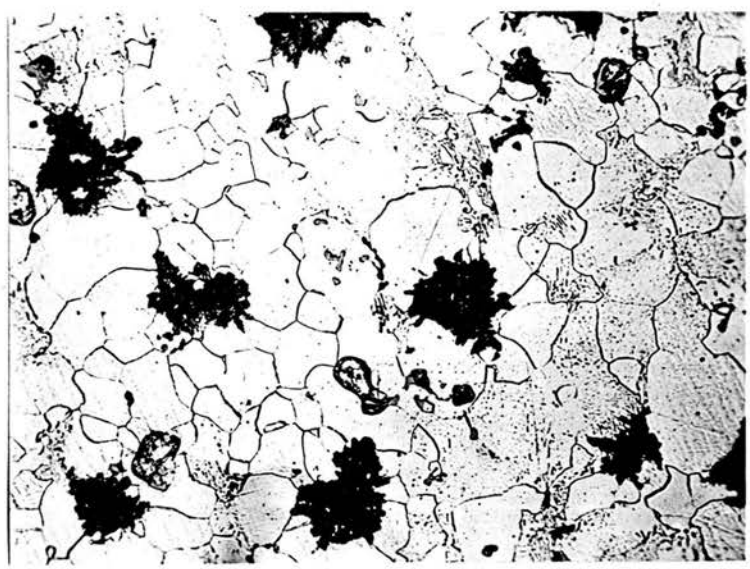
Etched 8 sec. 4 % nital



Mag. 50X

Fig. 2a

Group 2 Specimen a



Mag. 250X

Fig. 2b

Group 2 Specimen a

Etched 8 sec. 4 % nital

Fig. 3a Mag. 50X

Group 3 Specimen b

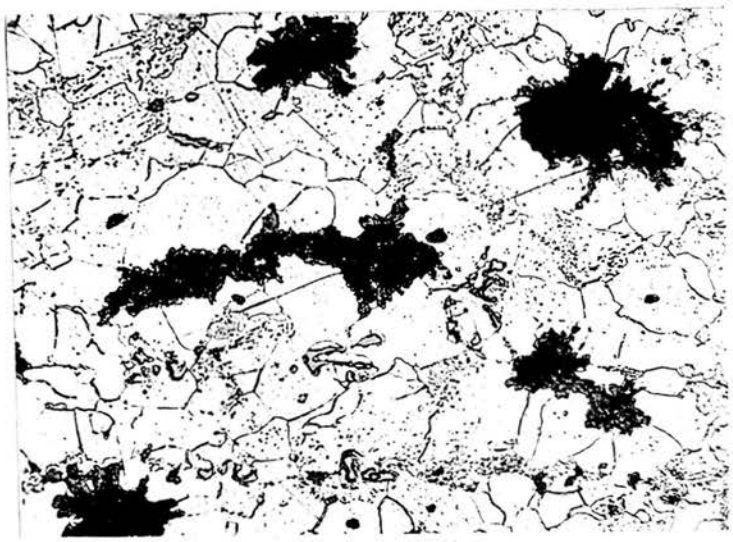


Fig. 3b Mag. 250X

Group 3 Specimen b

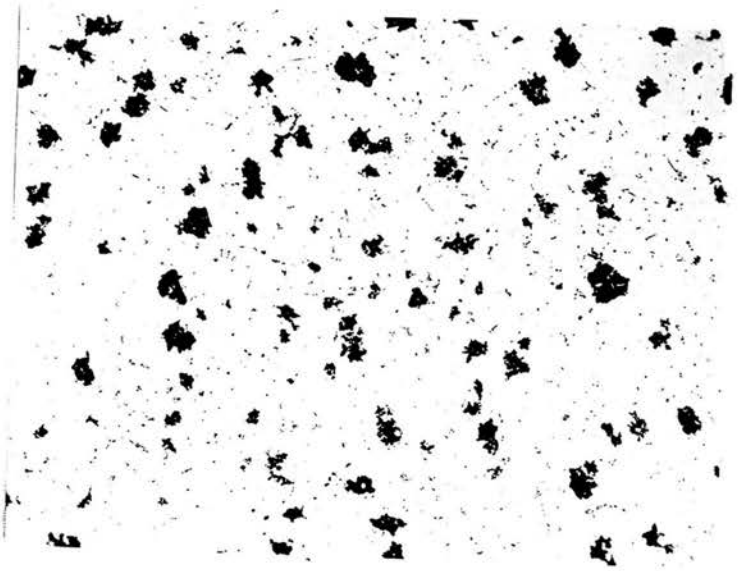


Fig. 4a Mag. 50X

Group 4 Specimen a

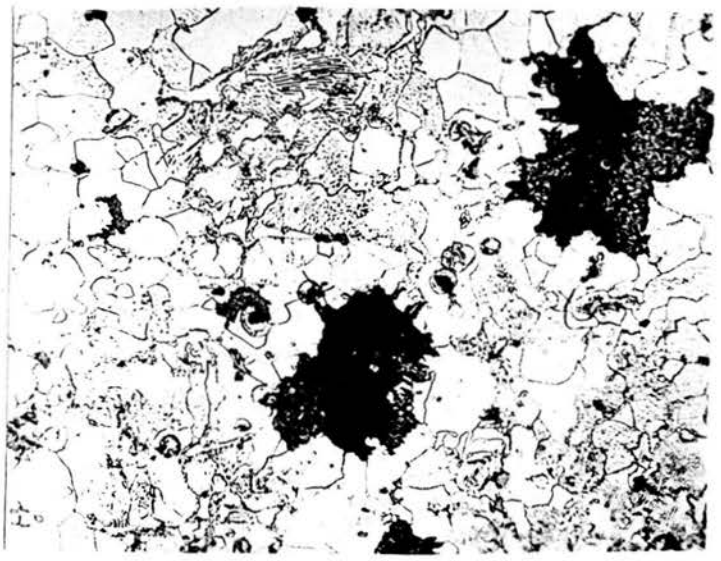


Fig. 4b Mag. 250X

Group 4 Specimen a

Etched 8 sec. 4% Nital

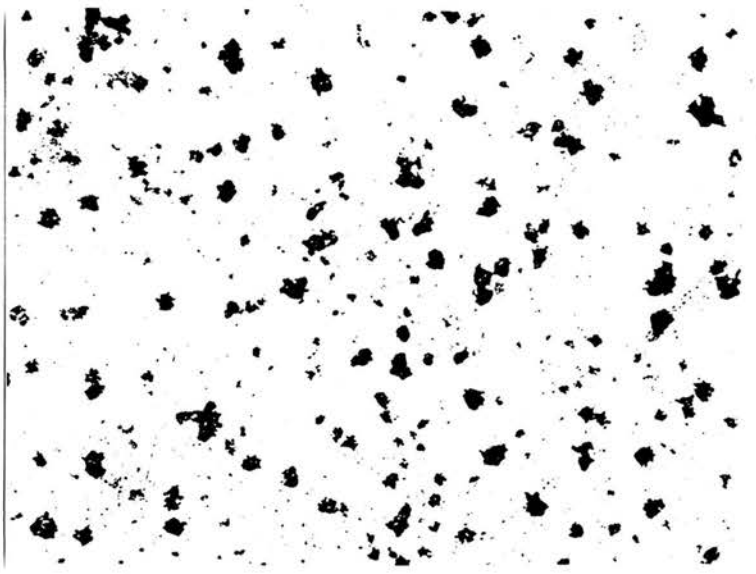


Fig. 5a

Mag. 50X

Group 5 Specimen b

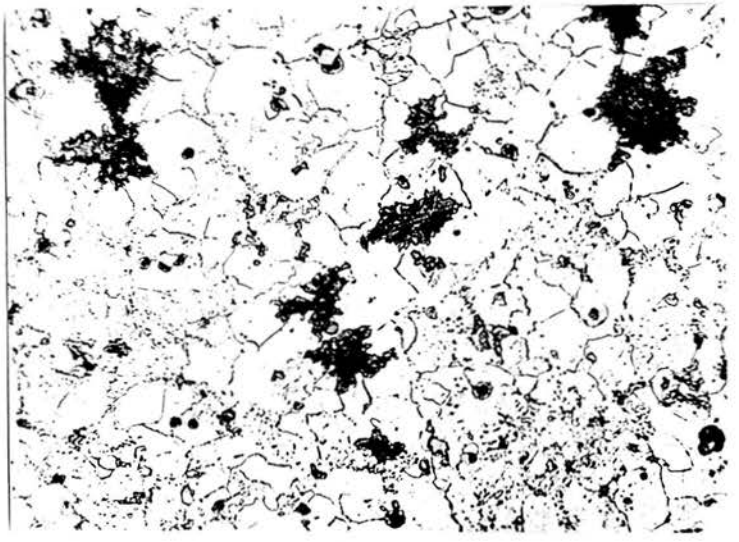


Fig. 5b

Mag. 250X

Group 5 Specimen b

Etched 8 sec. 4% Nital

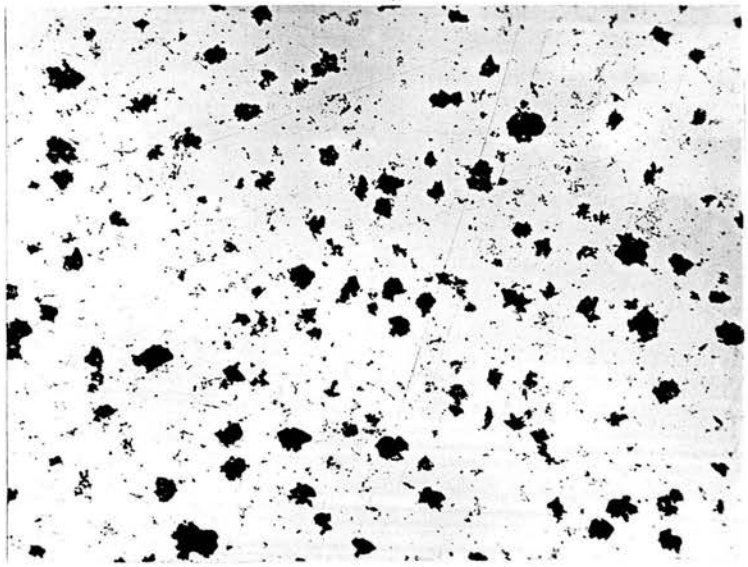


Fig. 6a

Mag. 50X

Group 6 Specimen a

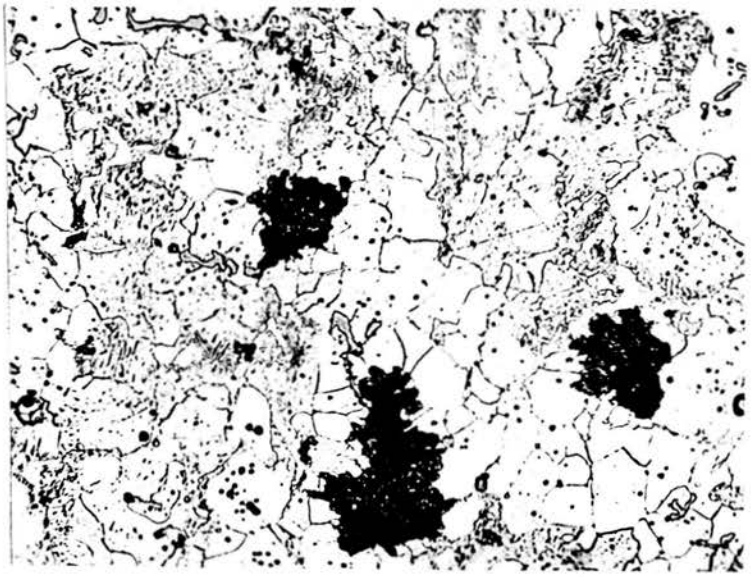


Fig. 6b

Mag. 250X

Group 6 Specimen a

Etched 8 sec. 4% Nital

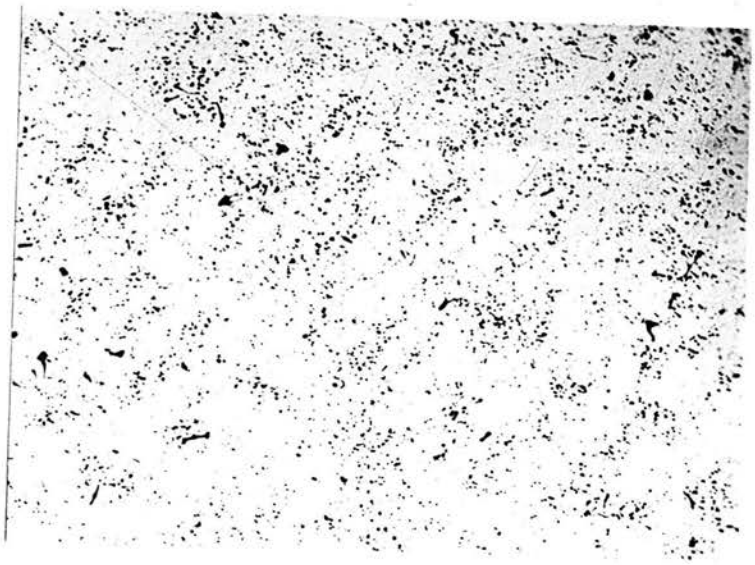


Fig. 7a Mag. 50X

Group 7 Specimen a

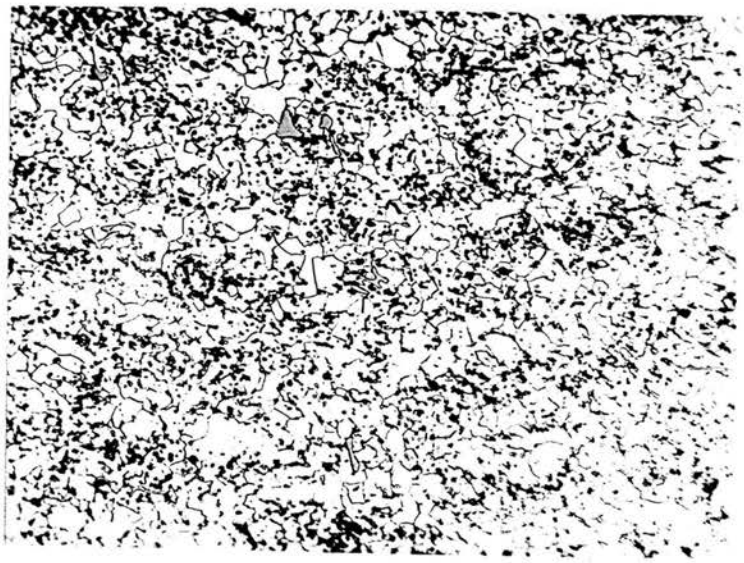


Fig. 7b Mag. 250X

Group 7 Specimen a

Etched 8 sec. 4% Nital

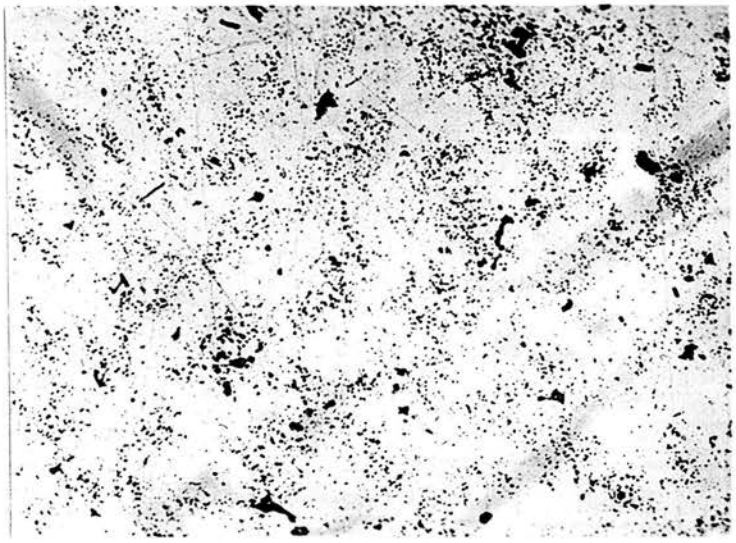


Fig. 8a

Mag. 50X

Group 8 Specimen a

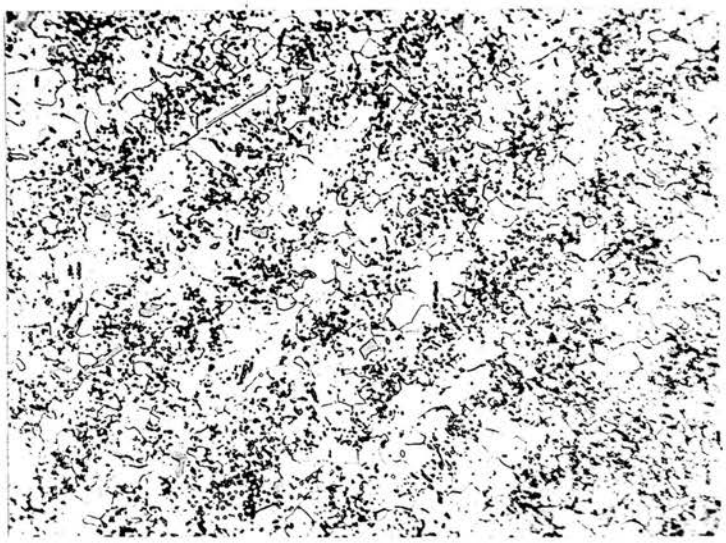


Fig. 8b

Mag. 250X

Group 8 Specimen a

Etched 8 sec. 4% Nital

Fig. 9a

Mag. 50X

Group 9 Specimen a

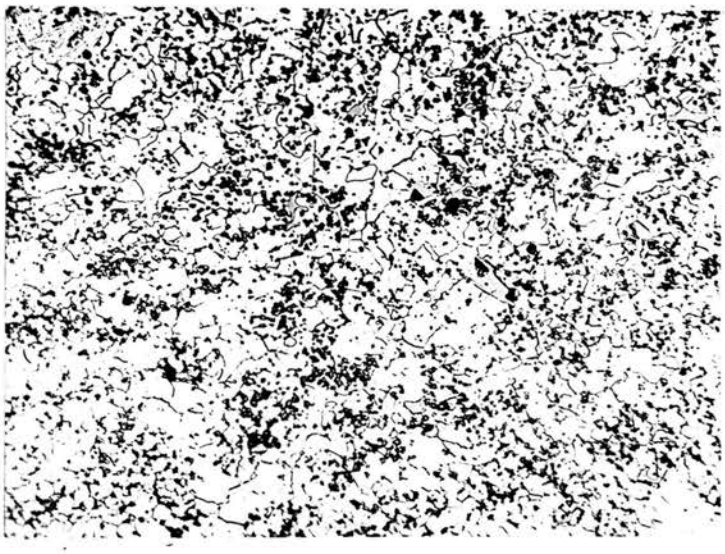


Fig. 9b

Mag. 250X

Group 9 Specimen a

Etched 8 sec. 4% Nital

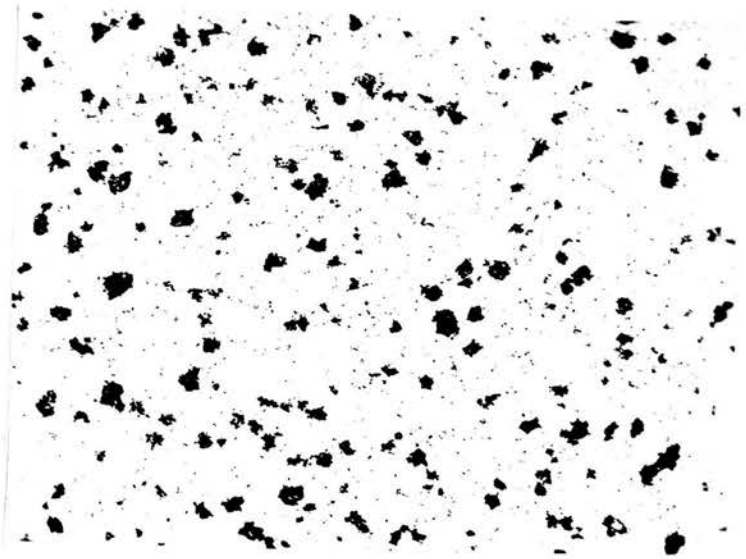


Fig. 10a

Mag. 50X

Group 10 Specimen a

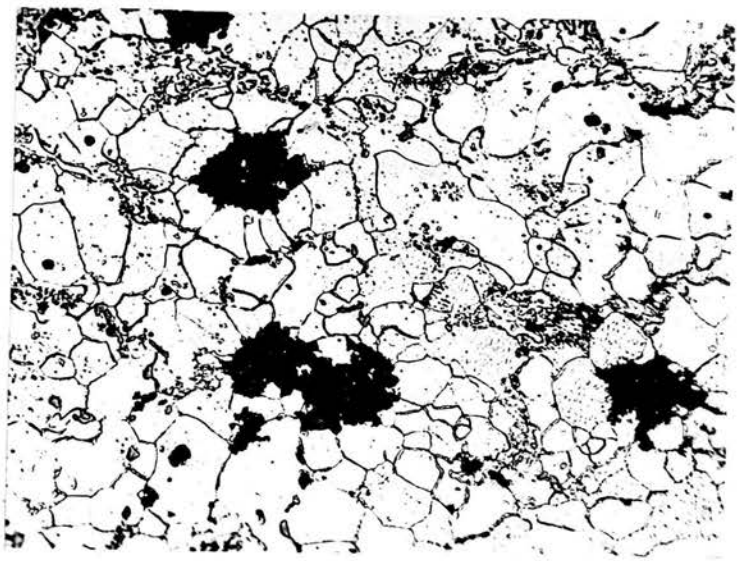


Fig. 10b

Mag. 250X

Group 10 Specimen a

Etched 8 sec. 4° Nital

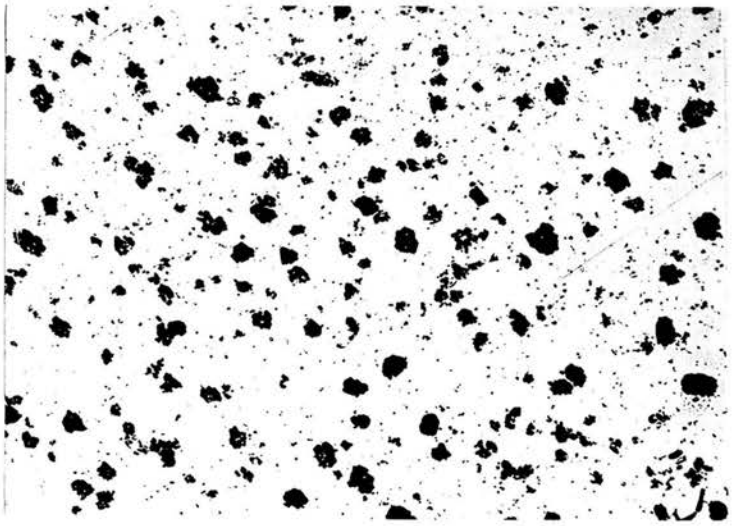


Fig. 11a Mag. 50X

Group 11 Specimen b

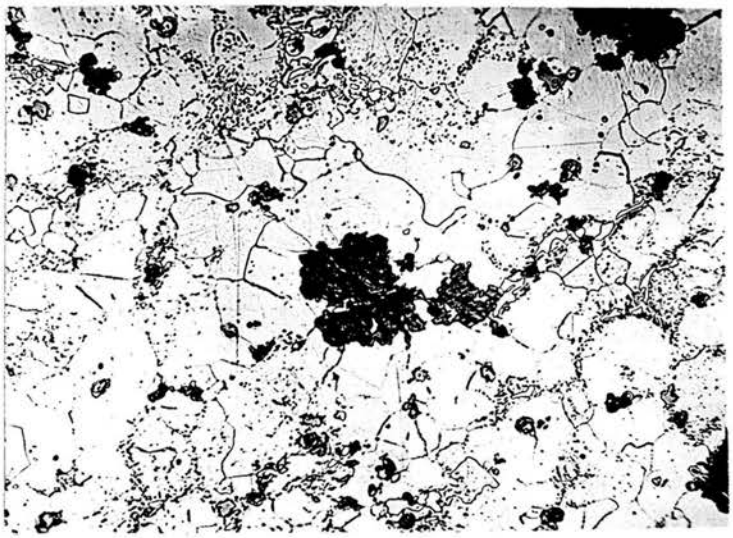


Fig. 11b Mag. 250X

Group 11 Specimen b

Etched 8 sec. 4% Nital

Fig. 12a

Mag. 50X

Group 12 Specimen a

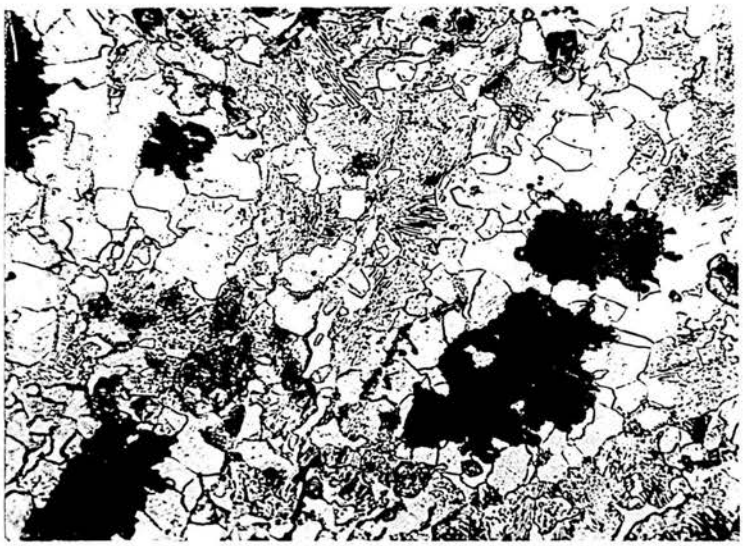


Fig. 12b

Mag. 250X

Group 12 Specimen a

Etched 8 sec. 4% Nital

Fig. 13a Mag. 50X

Group 13 Specimen a

Unetched

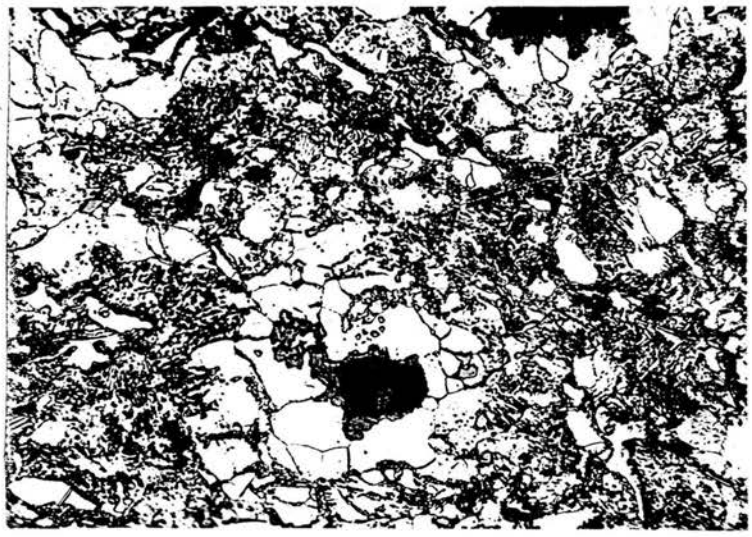


Fig. 13b Mag. 250X

Group 13 Specimen a

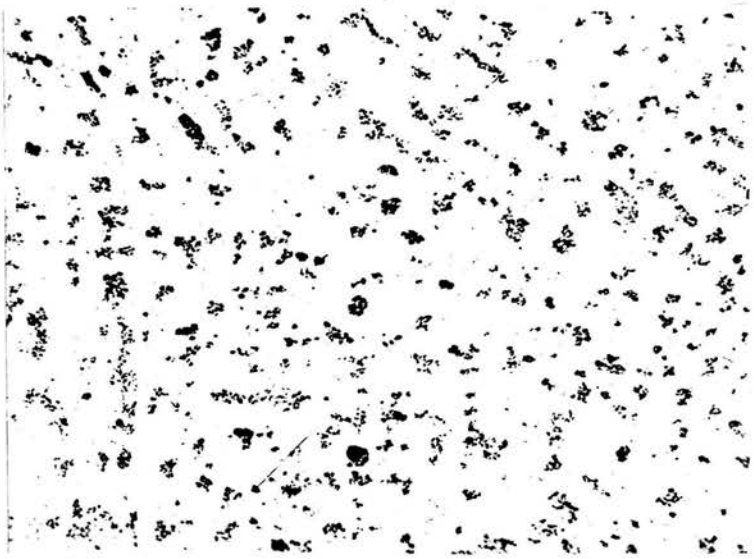


Fig. 14a

Mag. 50X

Group 14 Specimen c

Unetched

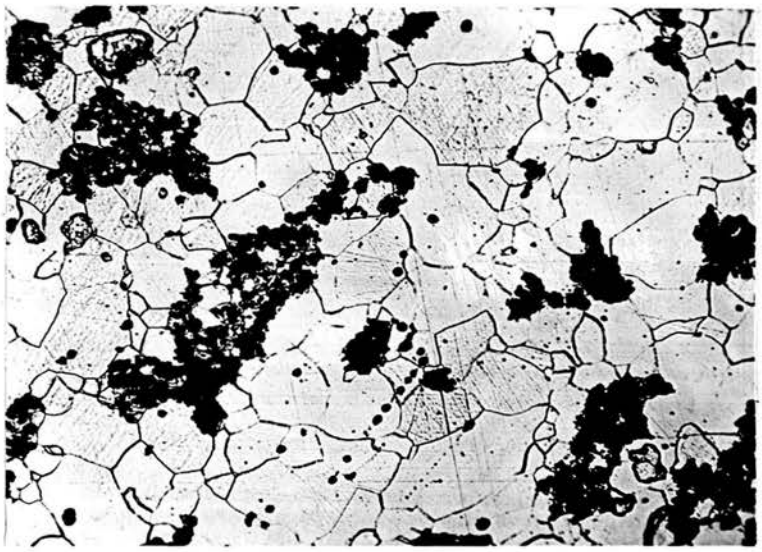


Fig. 14b

Mag. 250X

Group 14 Speciman c

Etched 8 sec. 4% Nital.

The character of the graphite was especially noted. Each nodule was compact and tending to approach a spherical shape. There were few sprawly nodules, and none of the expansive finger types seen in some malleable irons appear to be present. It should be noted that in each specimen there existed nodules of varying sizes. It is believed by the writer that this relative size range, and percentage of each size present is of more importance than the actual nodule number; and this relative size, and size range should perhaps be used rather than nodule number when comparing the mechanical and physical properties of malleable iron.

As the quenching rate increased for each group, the nodule size became smaller. Rates greater than 0.14 resulted in fine temper carbon nodules in what appeared to be a characteristic dendritic pattern. (Fig. 7b, 8b, 9b and 14b). DeSy [38] reported some photomicrographs of nodular iron

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(38) DeSy, Albert, Spherulite Formation in Nodular Cast Iron, Metals Progress, p. 798, June 1951.

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quenched early in the solidification stage, and, in each case, the nodules were present in the dendritic pattern.

## PHYSICAL PROPERTIES OF GROUPS 1 TO 14

Group 1: The base "as cast" white iron of Group 1 would pass the ASTM standard grade 32510. This grade should have a minimum tensile strength of 50,000 psi, minimum yield point of 32,500 psi, and an elongation of 10% or better. If Table B is consulted, Group 1 has an average tensile strength of 56,300 psi, yield strength of 40,300, and an elongation of 10.8%. The Vickers hardness is 129, or approximately 115 Brinell. The restricted elongation is due perhaps to the 5-10% pearlite, for the grain size is relatively large, and the nodule count is between 65 and 90 depending on the cross section. If both the stressed and relieved coercive force values of a representative sample from group one are considered, the bars ruptured at full stress, and did not fail due to possible casting defects.

Group 2: The pre-treatment (Pb bath at 800 F) given group 2 did increase the nodule count by 50%. (120 nodules per sq. mm.) The results of this treatment was evident only in an increase in elongation of approximately 2%. Tensile and yield strengths remained the same. This increase in elongation is probably due to the increase in ferrite grain size.

Groups 3, 4, and 5: Groups 3, 4, and 5 represent various air treatments (furnace cool, air cool, and air stream quench). Theoretically it should be expected, that the nodule count will increase, elongation decrease, and yield and tensile strengths increase progressively from group 3 to 5.

The experimental results indicate this is generally true,

with the exception of group 3, which has a higher tensile strength than would be predicted from the nodule count. The estimated pearlite and combined carbon are higher than the group 4, hence this may complicate the above relationships. Relative coercive force values indicate the possibility that group 4 did not rupture at the ultimate stress.

Groups 6, 7, 8, and 9: Groups 6, 7, 8 and 9 were subjected to water quenching pretreatments. Each at a known rate relative to group 8. Rates of quench were 0.044, 0.17, 1.0, and 2.0 respectively. These pretreatments resulted in nodule counts of 100, 4000, 8000, and 10,000 plus, nodules per square mm. (If any further work is to be done, pretreatment quenching rates from 0.044 to 0.17 should be used.) As the nodule number increased, the tensile strength increased, and the elongation dropped to zero. The fracture of group 8 and 9 bars was silky and smooth indicating a brittle fracture.

As was noted under "Tensile Strength" groups 8 and 9 resulted in erratic tensile values. The tensile strengths for 8a-37,200, 8b-59,200, 8c-35,000, 9a-63,350, 9b-38,000, 9c-62,000 psi. The rate of loading for specimen 8b, 9a, and 9c was 2,500 lbs/min. for specimens 8a, 8c, and 9b was 1000 lbs/min. hence the rate change is 2.5. Coercive force values of the stressed and relieved specimens from the 3/8 inch section indicate specimens 8a, 8c, and 9b did not rupture at full stress, but at approximately 60% of the ultimate value. If the stressed and relieved values for 7a, 8a, and 9a are con-

sidered, the differential coercive force is approximately 7a-2.63, 8a-0.46, 9a-2.60. Specimen 8a ruptured at a lower stress than 7a or 9a, since coercive force is proportional to stress. (See Graph 10, page 27.)

Groups 10, 11, 12, and 13: Groups 10, 11, 12 and 13 were subjected to quenching in the same medium, but the temperature to which the bars were heated before quenching was varied. Group 6 should also be considered here, for water at 212 F was used as its quenching medium. The heating temperatures (F) were as follows, Gp. 10-1700, Gp. 11-1600, Gp. 6-1500, Gp. 12-1360 (approximately $A_1$), Gp. 13-1200 (below the $A_1$). The nodule counts were 180, 135, 100, 75, and 80 nodules per sq. mm respectively. The nodule count is directly proportional to the temperature to which the bars were subjected. Group 13 is the exception. This group was held below the $A_1$, and resulted in a nodule count approximately the same as the base bars of Group 1.

The tensile strength of group 10 bars was not as great as would be expected, due to the character of the nodules. (See Fig. 10a and 10b, page 63.) It was noted that these nodules tended to be more open and sprawling than others in this series. This is believed to be due to the higher quenching temperature.

Group 14: Group 14 was pretreated in an oil quench before annealing. The resulting nodule count was 250 nodules per sq. mm. The tensile strength for the bars of this group is believed to be low, this was substantiated by the coercive force tests. Note the small differential from stressed to relieved.

Also if Fig. 14a and b are considered, it can be seen that the nodules of Specimen 14c tend to form definite patterns, and groupings. At low power this pattern appears to be dendritic in form. It readily can be seen that these stringers could present planes of weakness in the cross section (just as graphite flakes do in grey iron). If this were so, the elongation should be low. The elongation for the test bars of the group are as follows, 6.5%, 4.0%, and 8.5%.

From the preceding discussion of specific physical and mechanical properties of the various groups, it is evident that temper carbon nodule number is a factor in determining the physical and mechanical properties of malleable iron, however it is not the only controlling factor. Ferrite grain size and percent pearlite must also be considered.

As the temper carbon nodule number increases, the ferrite grain size decreases. It has been reported, (39 fer-

(39) Edwards, C.A., and Feil, L.B., Ferrite, Journal Iron Steel Inst., Vol. 112, p. 129, 1925.

rite tensile strength may vary from 20,000 psi for a single grain, to 42,500 psi for very fine ferrite (200 grains per sq. mm). A difference of 20,000 psi due to grain size alone.

As nodule number decreases, the rate of graphitization in annealing also decreases, hence the percentage of pearlite may tend to increase if the annealing cycle is held constant.

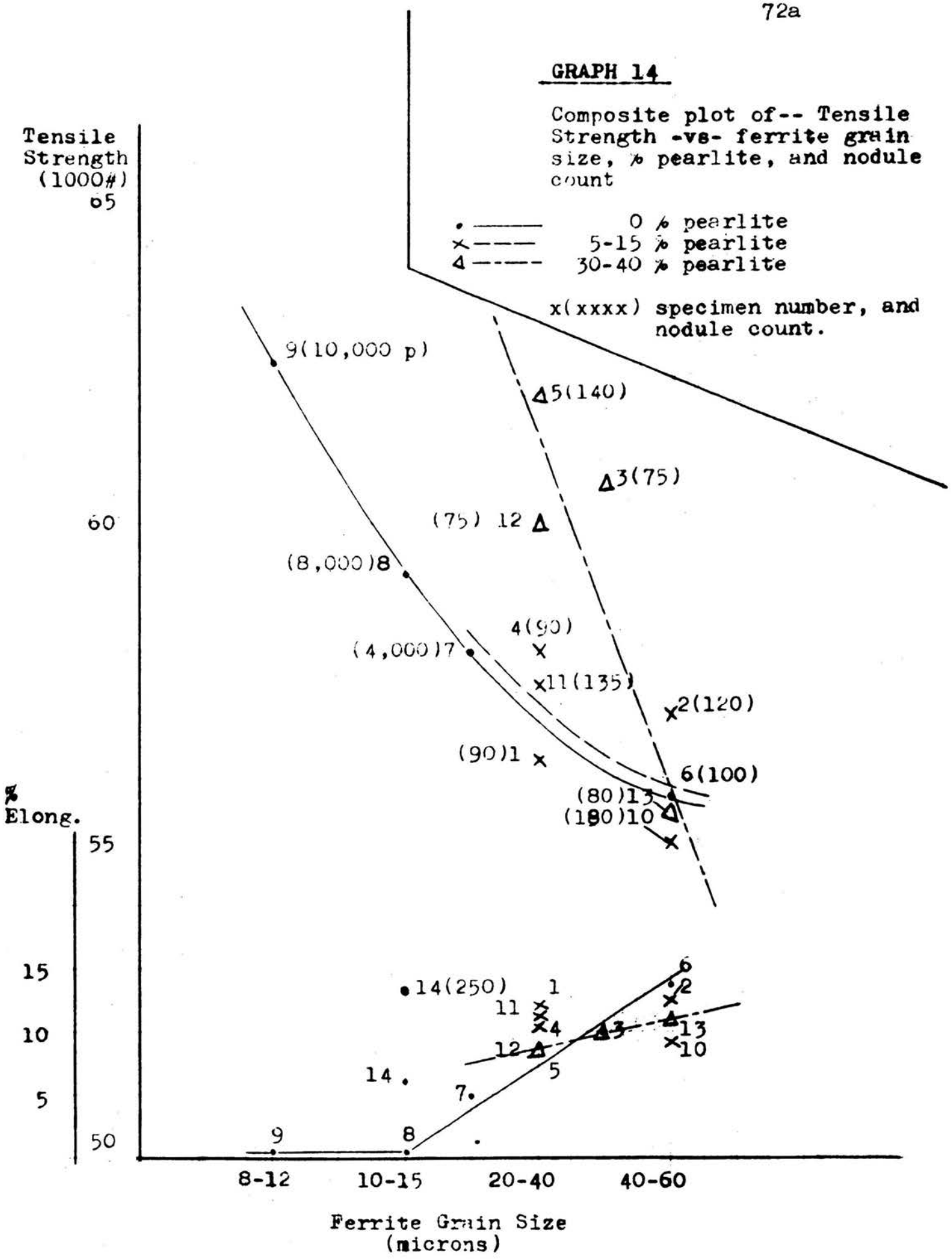
Graph 14: In an attempt to correlate these various factors, Graph 14 was prepared from data as presented in Table B.

**GRAPH 14**

Composite plot of-- Tensile Strength -vs- ferrite grain size, % pearlite, and nodule count

•——— 0 % pearlite
×---- 5-15 % pearlite
Δ----- 30-40 % pearlite

x(xxxx) specimen number, and nodule count.



It is a composite plot of tensile strength and elongation versus the ferrite grain size and nodule number at different percentages of pearlite.

From this experimental plot, the following relationships were observed: considering 0 % pearlite, (100% ferrite) -- as the temper carbon nodule number increases, the tensile strength increases and the elongation decreases. 8,000 nodules per sq. mm results in an increase of 4,000 psi in tensile strength, however the elongation is decreased to zero. 10,000 nodules per sq. mm causes an increase in tensile strength of 7,000 psi. It should be noted that in each case the ferrite grain size decreased as the nodule number increased. Nodule count appeared to be the prime factor for the increase of strength, with the ferrite grain size exerting a secondary influence. This held for 100% ferrite only.

With 5 to 15% pearlite, nodule number did not appear to be the controlling factor, but the ferrite grain size would seem to have had a greater influence on the strength. Elongation was approximately constant for the nodule range tested (90 to 180 nodules per sq. mm)

The 30 to 40 % pearlite range showed an increase in strength proportional to the ferrite grain size, and not the nodule number. Specimens 3 and 5 indicated there was a definite increase in strength due to nodule count, but Specimens 12 and 3 tended to disprove this assumption. One possible explanation of this was found in reference to Joly's [40] symposium

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(40) Joly, G., op. cit., p. 12.

work on malleable iron (See graph No. 2, page 14). Joly tested 82 bars in each series, and found there was a spread of tensile strengths for the overall test group. Note the overlap of the low nodule count group into the range of the high nodule count group. Hence it would be possible to break a few bars in each series, and obtain the same tensile strengths for one having few nodules as for one having many nodules. It is even probable, that the tensile strength of the low nodule count series could be greater than for the higher nodule count series.

It was noticed in Graph 14, that there was a possible overlap of test specimens containing 90, 135, and 4000 temper carbon nodules per sq. mm. Also two groups, (one, Gp. 5-140 nodules per sq. mm, and the other, Gp. 9-10,000 nodules per sq. mm) represented the maximum tensile strengths obtained in this investigation. Group 5 had 30 to 40 % pearlite, group 9 was 100 % ferritic.

The percent elongation of the 30 to 40 % pearlite series decreased as the ferrite grain size decreases, and not in relation to the relative nodule number.

The position of Group 14 (250 nodules per sq. mm) should be noted in that it is unique in exhibiting low tensile strength. Considering the nodule count, and ferrite grain size, this group should have ruptured about 57 to 58,000 psi.

## CONCLUSIONS

1. The temper carbon nodule number may be varied by thermal pretreatment. A variation of nodule number has been achieved in this investigation, with a range of from 66 to 250, and 4,000 to 10,000 temper carbon nodules being used in the experiment.

2. There exists a definite relationship between temper carbon nodule number, ferrite grain size, tensile strength and elongation. This relationship is most pronounced in 100% ferritic malleable iron. Temper carbon nodule number does not appear to be a prime factor in controlling the tensile strength as the percent pearlite in the matrix is increased.

3. This investigation has shown that it is more reliable to use temper carbon nodule size, and size distribution, instead of Temper carbon nodule number when relating physical and mechanical properties.

4. Coercive force values have successfully been used to indicate strength (tensile strength) correlations between the various groups of malleable iron. It is possible to predict if the tensile strength, found in testing a set of tensile test bars is the ultimate stress which the metal can inherently withstand, the failure may have been caused by a mechanical defect. This property has been found to be insensitive to the matrix structure of the malleable cast iron.

5. Coercive force values have also been used to estimate the temper carbon nodule count in a group of test specimens, varying in count from 66 to 250 nodules per sq. mm. Where

appears to be a discontinuity in the coercive force values relative to nodule count at approximately 250 to 300 temper carbon nodules per sq. mm. This relationship has been found to be sensitive to matrix structure of the malleable iron.

5. It is believed the quenching media used introduced a variable into this investigation. Any further work should be accomplished by using the same type or class of media.

6. Forty two bars were tested, and each group contained but three bars. The limitations due to this small number is fully realized. It is believed more work will have to be done to substantiate the coercive force - temper carbon nodule number relationship.

## SUGGESTIONS FOR FURTHER WORK

1. This work should possibly be repeated using salt baths at various temperatures as the quenching medium, in this way the different quenching rates will be achieved.
2. A uniform set of specimens approximately 0.35 inches in diameter, and 1.475 inches long should be obtained from the Chicago Malleable Co. These should be pretreated to vary the temper carbon nodule count, then annealed to 100% ferrite. Coercive force readings could be obtained from these specimens, with no special machining. In this way minimum stress will be present, and a true relation between the temper carbon nodule count and coercive force could be had.
3. Further work should be done varying the percent pearlite present, for no commercial malleable iron castings are entirely free from it, due to variation in size of section.
4. If possible the physical properties of identical matrix structures of a malleable iron, and a plain carbon steel should be compared. Using perhaps 100% ferrite, 5-10% pearlite, 30-40% pearlite, and 75 to 100% pearlite.

# APPENDIX

## Polishing Technique using diamond dust

Diamond dust in a vehicle can be used in the metallographic laboratory in place of the other common polishing compounds without the usual slowness and dirt that accompanies carborundum and alumina. Its most important feature in polishing duplex structures such as malleable iron, grey iron, etc. is that when using a short nap cloth, it does not tend to pull the graphite, or round or flow the edge of the graphitemetal interface.

## Procedure

First step: Prepare a flat surface on the specimen with coarse paper or wheel. The writer used 180 grit wheel, and papers 1, 2, and 0. The usual precautions, such as washing the specimen and hands between papers, should be observed. The most important point, is light pressure at all times. Do not push the specimen into the wheel or papers.

Second step: (First diamond wheel). Be sure the specimen has been washed well before starting on the diamond wheels.

This wheel is to have a canvas cloth which has been preshrunk to fit. To this wheel put a small amount (in small amounts around the outside quarter) of the red diamond dust, No. 30. (After the wheel has been coated well the first time less dust will be required for the next specimens.) Spread the dust with the finger and turn the wheel at its lowest speed. Apply the specimen at 90 degrees from the direction which it was previously polished on the paper or initial

grinder. Apply a steady force, examining the surface until the specimen becomes shiny, and no more right angle scratches are present. Then add small amounts of water until the specimen becomes more shiny (polished).

W ^H THOROUGHLY WITH ALCOHOL BEFORE GOING TO THE NEXT WHEEL, TO REMOVE ALL TRACES OF THE GREASE FROM THE VEHICLE IN THE DIAMOND DUST.

Third step: (Second wheel) This wheel should have a pre-shrunk microcloth, upon which is put a small amount of the yellow diamond dust, No. 3. Polish at 90 degrees from the first sheel and continue in the same manner as in the second step, having the wheel at the lowest speed. WASH THOROUGLY, ETC.

Fourth step: (Third wheel) This wheel also has a pre-shrunk microcloth, miracloth, or an airplane silk cloth, upon which has been put a very small amount of the Ivory diamond dust, No. 1. Polish in a direction 90 degrees from the second wheel and continue in the same manner as before having the wheel at its lowest speed. Polish until a mirror finish has been obtained. Very little pressure is needed on this final step.

Some Pertinent Notes

Some specimens may be polished after the third step; if so, do not use the fourth step.

If possible keep the cloths damp but not wet during polishing.

Very little time is needed on each wheel, to accomplish the polishing.

Of the three dusts, the Yellow No. 3 is the working dust, and careful attention to this wheel (polishing technique) will yield excellent results.

Diamet-Hyprez fluid or Carbon Tetrachloride may be used as a lubricant.

Other lubricants are olive oil, peanut oil, oil of lavender, and mineral oils of different viscosities.

Diamond-Hyprez dusts are suspended in mineral oils.

The diamond dusts range in size from 120 to 0.5 microns.

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