AN INVESTIGATION TO DETERMINE THE MANNER OF SOLIDIFICATION AND TRANSFORMATION OF NODULAR CAST IRON

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Douglas J. Harvey

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This is to certify that the

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An Investigation to Determine the Manner of Solidification and Transformation of Nodular Cast Iron

By

Douglas J. Harvey

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INTRODUCTION

The recent development of nodular iron has been the most important discovery in the field of cast ferrous metals since Seth Boyden first produced black heart malleable iron in 1826[1]. Although nodular iron is still in the development stage many tons are being produced every day.

Gray cast iron is essentially a ferrous alloy with 2.6-3.75% carbon and 1.25-2.75% silicon. During solidification most of the carbon leaves solution and appears in the cast metal as graphite flake inclusions. These inclusions break up the continuity of gray cast iron and account for its brittle and nonductile properties.

In certain composition ranges castings with thin sections can be made with all their carbon in the combined form. These white iron castings can be made into malleable iron by a long and costly heat treatment. The malleable iron that is produced in this country has its free carbon in small clumps. These clumps of graphite, commonly called temper carbon, do not break up the continuity of the malleable iron as severely as the graphite flakes in gray cast iron. Thus we see how a difference in graphite shape changes the properties from brittle to ductile and increases the tensile strength two to

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1. Simpson, Bruce, L., Development of the Metal Castings Industry, American Foundrymen's Association, Chicago, Illinois, 1948, p. 196.

three times.

Nodular iron has its graphite in nodules that appear very similar to the clumps of temper carbon in malleable iron. The shape of the graphite in nodular iron is responsible for its high strength and good ductility. Nodular iron can be produced from a base iron in the common ranges for malleable iron, gray iron, or even pig iron. This new material is made by additions of magnesium to the molten base iron. The magnesium addition is followed by inoculation with ferrosilicon. Cast iron so treated solidifies as nodular cast iron and needs no costly heat treatment.

The first hint that an iron could be cast with its graphite in nodular form came in 1930. Von Keil[2] published he had produced cast iron with nodular graphite. The methods he used were essentially the same as those used today. Additions of magnesium were made to the molten metal followed by inoculation with silicon. The cast alloy which received this treatment had its graphite in nodular form. For some unknown reason this early reference to "as cast" nodular graphite escaped widespread attention.

There is no indication that anything more was done with nodular iron until 1947 when Morrogh and Williams[3] of the

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2. von Keil, O., Die Graphitbildung im Gusseisen., Archiv fur das Eisenhuttenwesen, Vol. 4, pp. 245-250, (November, 1930)
3. Morrogh, H. and Williams, W. J., "Graphite Formation in Cast Irons and in Nickel-Carbon and Cobalt-Carbon Alloys," Journal of the Iron and Steel Institute, vol. 155, pp. 321-370, (March, 1947).

British Cast Iron Research Association made public their work on graphite formation. This important work was carried on using nickel-carbon alloys which are analogous in their behavior to alloys of a composition in the cast iron range. They found that nodular graphite could be produced in these alloys under certain conditions.

Morrogh and Williams[4] in a later work demonstrated they could consistantly produce nodular cast iron. This new material was produced by additions of cerium to an iron that was hypereutectic.

On May 7, 1948 at the American Foundryman Society meeting in Philadelphia Thomas H. Wickenden[5] of the International Nickel Co. announced that his company had produced nodular iron using additions of magnesium.

The first work on magnesium additions was published in February, 1949 by C. K. Donoho[6] of the American Cast Iron Pipe Co.

During the period 1949-1951 many articles have been published on nodular iron. However very few of these publica-

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4. Morrogh, H. and Williams, W. J., "The Production of Nodular Graphite Structures in Cast Iron," Iron and Steel, vol. 158, pp. 306-314 (1948).
5. Discussion by Thomas H. Wickenden, International Nickel Co., Inc., of the paper entitled "Production of Nodular Structure in Cast Iron", by H. Morrogh, presented at the May 1948 annual meeting of the American Foundrymen's Society.
6. Donoho, C. K., "Producing Nodular Graphite with Magnesium", American Foundryman, vol. 15, pp. 30-35 (February, 1949).

tions have any information on the mechanism of nodular graphite formation. Why, after certain additions, does the graphite form nodules instead of the familiar flakes? How are these nodules formed? When during the process of solidification do the nodules of graphite make their appearance? No complete answer to any of these important questions has been published.

SURVEY OF WORK ON GRAPHITE FORMATION

Howe[7] with a knowledge of the stable and metastable iron iron-carbide equilibrium diagrams described the solidification of cast iron. His view was that primary dendrites of austenite are formed first in hypoeutectic alloys while in hypereutectic alloys the primary dendrites are iron-carbide. The freezing of the primary structure is followed by the solidification of the eutectic liquid. Howe's concept of solidification was in perfect agreement with the observed structure of white iron. The difference in white iron and gray iron were explained by Howe in terms of the stable and metastable equilibria. He gave no hint as to the manner or mode of the formation of graphite in gray cast iron.

The mechanism and manner of graphite formation in gray iron has been and still is a controversial issue. Hurst[8] writing in 1926 gave arguments for two possible answers to the graphite problem: solidification of the primary followed by the formation of a graphite austenite eutectic; or solidification of the primary dendrites followed by the formation of an austenite cementite eutectic and then an ensuing reaction in which graphite is formed. Hurst maintained at

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7. Howe, H. M., The Metallography of Steel and Cast Iron, McGraw-Hill Book Co., New York, N.Y., 1926.
8. Hurst, J. E., Metallurgy of Cast Iron, Isaac Pitman & Sons, London, 1926.

the time the information on the subject was unsatisfactory and there was a great need for more work on graphite formation in cast iron.

In 1936 work was carried on at Battelle Memorial Institute to determine the mechanism of graphite flake formation in gray cast iron. This work was carried on under the direction of Alfred Boyles. A full report of this work is contained in Boyles' book, The Structure of Cast Iron.[9] In this important study, samples of cast iron were allowed to cool slowly then quenched in water while in various stages of solidification. During slow cooling solidification proceeds according to the stable equilibria, but when the specimen is quenched solidification is so speeded up that the metastable system is more applicable. Therefore the specimen has a dual structure: the structure that forms during slow cooling and the structure that is formed during rapid cooling when the specimen is quenched. By studying the slow cooled structure in several specimens which were quenched at various stages during solidification the sequence of events during solidification could be determined.

From his very excellent work Boyles draws the following conclusions. "Freezing begins with the formation of skeleton

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9. Boyles, Alfred, The Structure of Cast Iron, American Society for Metals, Cleveland, Ohio, 1947.

crystals or dendrites of primary austenite, followed by crystallization of eutectic liquid from independent centers within the interstices of the dendrites. This mode of solidification is common to many alloy systems in which a primary constituent and a eutectic occur. In the case of the iron-carbon-silicon alloy the graphite flakes come into existence during the freezing of the eutectic. They do not appear until the eutectic starts to freeze and once the eutectic is completely frozen the flake structure is established and does not change as the alloy cools down to room temperature."[10] Boyles goes further to say, "The flakes grow more or less radially from the crystallization centers of the eutectic, forming colonies composed of graphite flakes and austenite such as those shown in Figure (1.)."[11]

Very little has been published on nodular graphite formation theory. There is little or no experimental evidence to support the material that has been published.

In their work with cerium treated nodular iron Morrogh and Williams[12] plotted cooling curves of a nodular iron with the following composition:

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10. Boyles, Alfred, The Structure of Cast Iron, American Society for Metals, Cleveland, Ohio, 1947, p. 33.
11. Ibid.
12. Morrogh, H. and Williams, W. J., "The Production of Nodular Graphite Structures in Cast Iron," Journal of the Iron and Steel Institute, vol. 158, pp. 316-317 (March, 1948).

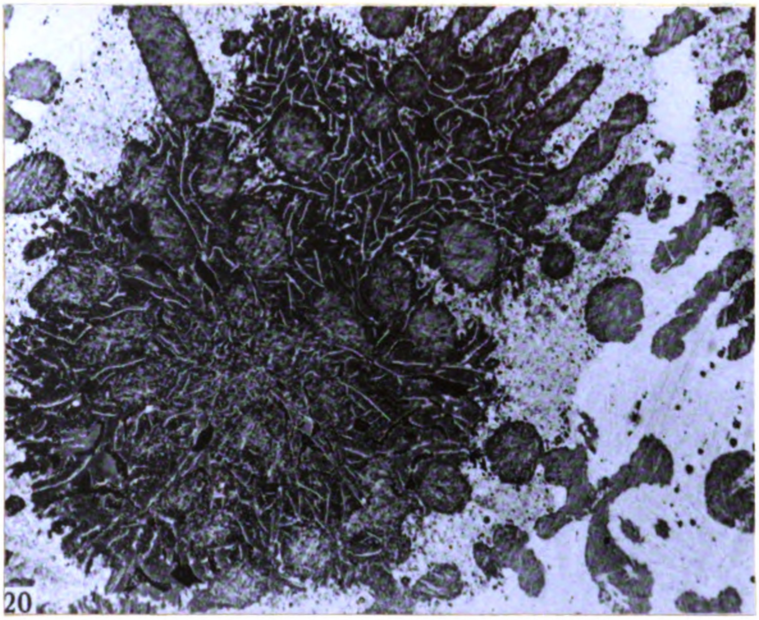

FIGURE 1. Graphite Flake Colony

total C.	Si.	Mn.	S.	P.	Ce.
3.91%	2.71%	.51%	.006%	.024%	.049%

The cooling curves they obtained were very similar to the ones shown in this work. The first arrest came at 2110°F. and the second came at 2090°F. A sample was quenched from a temperature of 1990°F. This sample was found to be completely graphitized and to have the same graphite structure as the slowly cooled specimen. A sample was also quenched just after complete solidification (just after the termination of the final arrest). This second sample was substantially white with some small and some large graphite spherulites. Morrogh and Williams said, "The large nodules were usually duplex in structure and were surrounded in every case by a zone free from carbide. This suggests that the hypereutectic spherulites form very early in solidification and, in their vicinity, graphitization proceeds very rapidly by deposition on the hypereutectic nuclei. Ingots quenched from temperatures between the two arrest points and also from above the first arrest point had structures (of) fine spherulites in a background of acicular white-iron eutectic."[13]

It must be kept in mind that Morrogh and Williams worked with an iron which was hypereutectic. It may be true that

13. Morrogh, H. and Williams, W. J., "The Production of Nodular Graphite Structures in Cast Iron", Journal of the Iron and Steel Institute, vol. 158, pp. 316-317 (March, 1948)

they found nodules of graphite forming before and during solidification in their irons that were hypereutectic. But it does not follow that hypoeutectic nodular irons are formed in a similar way.

Dr. Warren Larson[14] has made x-ray diffraction studies of graphite taken from various alloys. From his work with nodular graphite he has drawn the following conclusion. Nodules appear above and during the eutectic arrest. Dr. Larson also said that this early formation of nodular graphite during solidification explains the higher shrinkage which has been observed in nodular iron.

Rehder[15] from his work with nodular iron tentatively concludes: "1. Nodular graphite is the result of growth on specific nuclei. 2. There are radial and non-radial graphite types. 3. A nucleus exists in every nodule. 4. The nucleus is hard, relatively chemically inert. 5. The nucleus is hexagonal in crystal habit. 6. Size and size distribution of nodules is influenced by the nodulizing inoculant used. 7. Nodules contain silicon, iron, and titanium, with the existence of TiO_2 and Fe_2O_3 (this was) confirmed by x-ray diffraction. 8. The nucleus may be a carbide or a nitride."[16]

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14. Scobie, Herbert, F., "Nodular Iron Symposium Shows We Still Have a Long Way to Go", The American Foundryman, vol. 18, pp. 46-48 (October, 1950).
15. Ibid. pp. 47-48.
16. Ibid. pp. 47-48.

According to De Sy,[17] "Graphite nodules are born in supersaturated primary austenite." He states that his cooling curve studies correlate well with this conclusion. From highly hypereutectic nodular irons the nodules seem to form in the liquid. To back up this statement De Sy has photomicrographs from quenched specimens which show nodular graphite surrounded by martensite. In conclusion De Sy said, "Nodules are formed in the fully liquid or the fully solid, but never at a liquid-solid interface as in flake graphite."[18]

De Sy[19] also reports on two cast irons melted in the same manner in an induction furnace. The same raw material, the same magnesium treatment, and the same inoculation with ferosilicon was used in both heats. The only difference was in the carbon contents which were 2.02 and 3.56 per cent. According to De Sy in the low carbon heat, "The graphite appears in interdendritic strings of the mixed spherulitic and vermicular variety. This graphite issued from the liquid eutectic phase, or close to the eutectic composition, after the primary solidification of the dendrites of austenite."[20] The high carbon heat showed, "A structure of nodular cast iron which is as

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17. Scobie, Herbert F., "Nodular Iron Symposium Shows We Still Have a Long Way to Go", <u>The American Foundryman</u>, vol. 18, pp. 47. (October, 1950).
18. Ibid. p. 48.
19. De Sy, Albert, "Further Results of Belgian Nodular Cast Iron Research", <u>The American Foundryman</u>, vol. 17, pp. 75-83 (May, 1950)
20. Ibid. p. 77.

perfect as can be obtained. The composition of this cast iron comes close to the eutectic. Such a structure should be considered as having developed in the reverse(of the low carbon iron); the graphite is issued from a phase, probably a complex carbide, of primary precipitation. Such a structure is likely to be obtained only if we succeed in forcing the primary precipitation of almost all the carbon in excess of that soluble in the austenite." De Sy goes on to say, "To visualize formation of this structure we can start with a convenient composition and speculate on the concentration gradient of the silicon which is supposed to exist after the secondary inoculation with silicon."[21]

Summing up the findings on nodular graphite formation to date it has been found that:

1. Graphite nodules form in the liquid.
2. Graphite nodules form in the solid.
3. Graphite nodules form during solidification.
4. Graphite nodules form around small non-metallic inclusions.
5. Graphite nodules form around small metallic inclusions.
6. Graphite nodules form around graphite nuclei.
7. Graphite nodules have no nucleus.

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21. De Sy, Albert, "Further Results of Belgian Nodular Cast Iron Research", The American Foundryman, vol. 17, pp. 78.

8. There is a high degree of segregation of the nodules.

9. There is no segregation of the graphite nodules.

It is highly improbable that all of the above statements are true; in fact some are very contradictory.

Nodular iron is still definitely in the development stage. As more is learned about some of the underlying principals of graphitization in this new material the closer it will be to becoming an important engineering alloy.

PURPOSE AND SCOPE

1. To develop equipment for studying the graphitization of nodular iron.

2. To plot cooling curves of solidifying nodular iron.

3. To study the mode of graphite formation in a hypo-eutectic nodular iron.

In Boyles' work a small sample of cast iron of the desired chemical composition was melted in a crucible suspended in a vertical tube furnace.[22] After the sample had melted and reached the proper temperature the furnace power was turned off. When the furnace and sample cooled down to the required temperature the specimen and crucible were dropped from the furnace into a tank of water. This system works very good when gray cast iron is being investigated, but the making of nodular iron involves the addition of magnesium followed by an inoculation with ferrosilicon. Magnesium is a very active element and burns explosively in air. The boiling point of magnesium is lower than the super heat temperature of molten cast iron.[23] Addition of magnesium to molten iron in a tube furnace would be difficult and possibly dangerous.

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22. Boyles, Alfred, The Structure of Cast Iron, American Society for Metals, Cleveland, Ohio, 1947.
23. Boiling point of magnesium 2520°F. cast iron melts at 2110°F., but is superheated to 2815°F. before magnesium is added.

The effect of magnesium wears off in a short time if the iron is held in the molten state.[24] The cooling rate of the tube furnace and sample would be much too slow. For these reasons a method different than the one used by Boyles had to be developed.

It was decided early in this investigation that cooling curves of nodular iron must be plotted to determine the temperatures at which solidification begins and ends.

Because most of the nodular iron made using magnesium are hypoeutectic in composition it was decided to investigate an alloy that was hypoeutectic.

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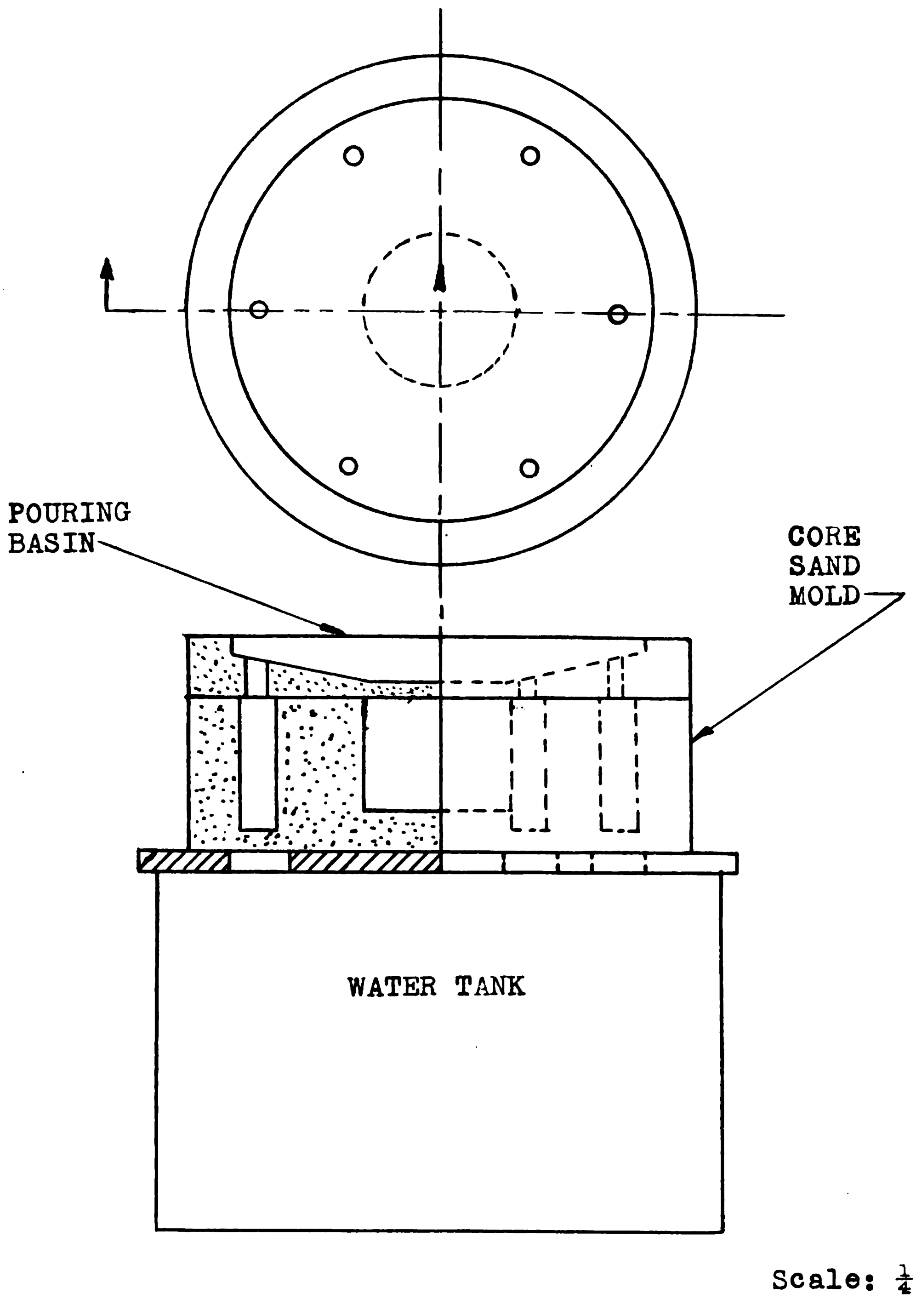
24. Donoho, C. K., "Producing Nodular Graphite with Magnesium", The American Foundryman, vol. 15 p. 32 (February, 1949).

PROCEDURE

The first equipment used is shown in Figure 2. This equipment consisted of a baked sand mold with six cavities connected by a common pouring basin. This mold was placed on an iron plate over a tank of water. Holes were drilled in the plate directly under each mold cavity. These holes allowed the specimens to be ejected into the water at any desired time. To keep the iron in the pouring basin from solidifying around the specimens, a drop out chamber was provided. As soon as the mold cavities and pouring basin were full the thin section of sand over the drop chamber was broken and the excess metal drained into it. This left six separate specimens ready to be quenched at the proper time.

Before the mold was poured a cromel vs. alumel thermocouple in a quartz protection tube was placed in one of the mold cavities. This thermocouple was connected to an indicating and recording potentiometer. This instrument produced a cooling curve of the solidifying specimen. It was assumed all of the specimens would cool at approximately the same rate. The object was to quench specimens at temperatures ranging from molten down to 1200^{o}F. By studying a microstructure from each of these six specimens, the sequence of events during graphitization might be determined.



Scale: $\frac{1}{4}$"=1"

FIGURE 2. Quenching Equipment

In heat No. N2 a steel and graphite charge was used. The iron was hypoeutectic having a carbon content of 3.5% and silicon at 1.96%.

Due to very rapid cooling of the specimens in the sand mold, temperature control was difficult. The samples were difficult to remove at the proper moment. Samples that were partly molten stuck on the end of the ejector rod. In spite of these difficulties some valuable information was obtained using this equipment.

Figure 3 is a photomicrograph taken of a sample that was quenched when partially molten. The primary dendrites of austenite are plainly visible. Centers of crystallization each solidifying around a nodule of graphite can be seen in the eutectic liquid. This microstructure shows that the nodules of graphite are formed very early during solidification.

Figure 4 is a microstructure of the sample quenched at near 1000°F. The size, shape, and distribution of the graphite nodules is very similar to those in Figure 3.

At this point some tentative conclusions can be drawn.

1. Freezing begins by solidification of the primary dendrites of austenite.

2. Graphite nodules appear in the eutectic liquid after the primary dendrites have formed.

3. The eutectic liquid freezes around the graphite nodules.

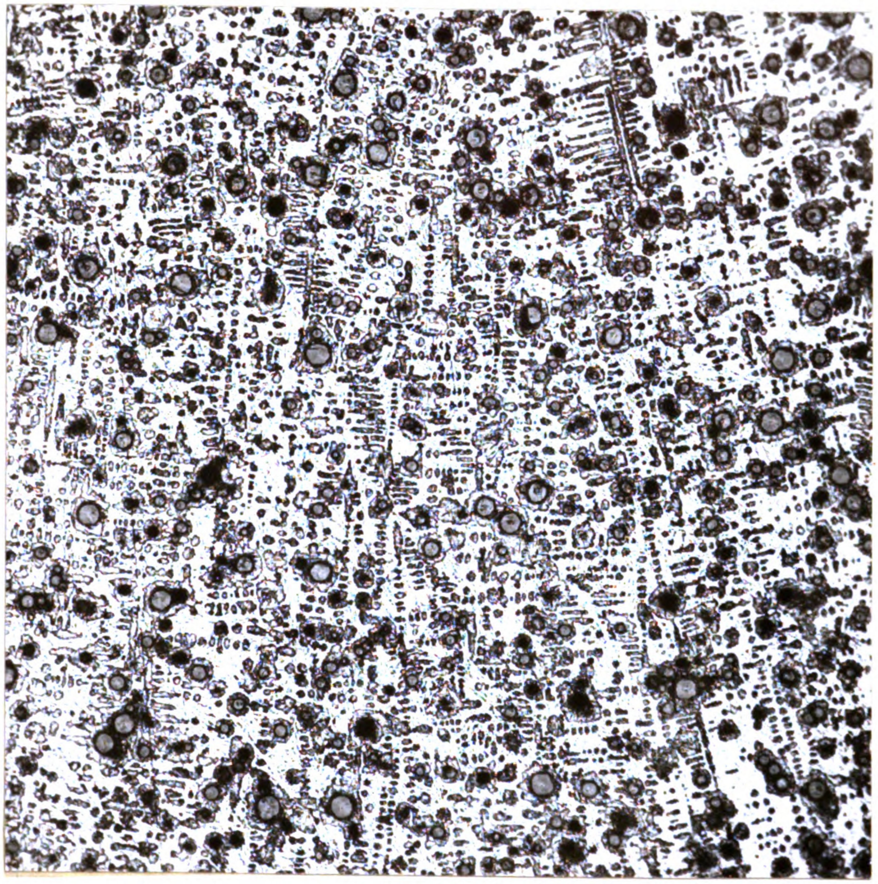


FIGURE 3. Heat No. N2

Treatment: Quenched when partially molten.

Magnification: 50 X

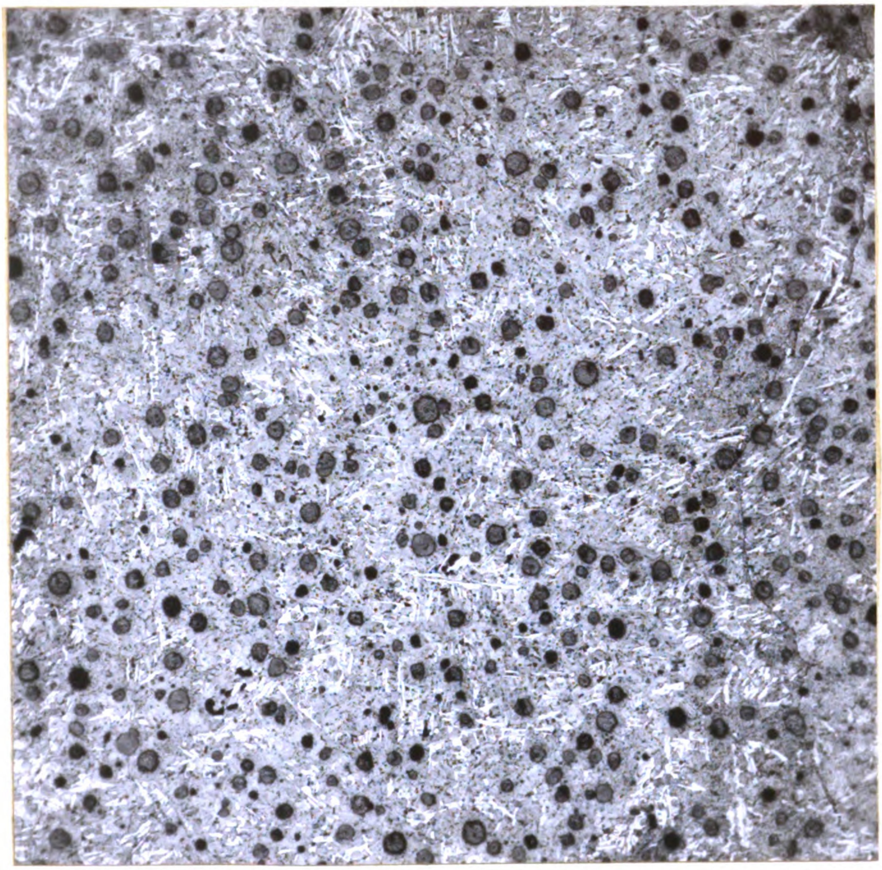


FIGURE 4. Heat No. N2

Treatment: Quenched from 1000 °F.

Magnification: 50 X

4. Very little graphite is formed after solidification is complete.

Before abandoning the dry sand mold equipment (Figure 2.) several unsuccessful attempts were made at reproducing the results obtained from heat No. N2.

The next attempt was made using a wedged shape casting. The plan was to quench the wedge when it had partly solidified. It was thought that the rapidly cooled casting would give a quenched structure of all phases of solidification. Some of the difficulties were that the tip of the wedge solidified so rapidly it was chilled white and it was difficult to tell how far solidification had progressed along the wedge. This made it difficult to know when to break the mold and quench the specimen. The two following difficulties made it next to impossible to gain information by this method: the light section of the casting that could be cooled rapidly by the quench solidified abnormally; the heavy section of the wedge that solidified normally was too large and transformation could not be arrested by action of the quench.

At this point it was clear just what the equipment needed should be able to do. A small sample of iron should be used so it could be quenched effectively. This small sample should be cooled slowly enough so that solidification would progress in a normal manner. The temperature of the sample should be accurately known at all times so the proper time to quench could be determined.

The equipment which was used successfully is pictured in Figure 5. It consists of a glow bar type tube furnace suspended vertically from a mono rail hoist. The position of the tube furnace could be changed by rolling the hoist along the rail. A 10 ml. alundum crucible supported by a cromel wire hanger was suspended in the tube furnace. The crucible hanger and a casting are shown in Figure 6. The cromel wire hanger was long enough to extend approximately two feet from the upper end of the tube. This allowed the crucible to be lowered into the induction furnace or quenching tank. A cromel vs. alumel thermocouple protected by a quartz tube was placed in the crucible. The thermocouple extention wires and their insulators were fastened to the crucible hanger wire.

The rapid recorder was assembled in the college shop and is very similar to the one used by Boyles in his work on inoculation[25]. The measuring part of the recorder consists of a Leads and Northrup portable potentiometer. A large pulley was substituted for the hand adjustment knob. The recording part consists of a chart driven by a synchronous motor drive. The pen of the recorder is connected to the pulley on the potentiometer by a wire. The position of the pen is very accurately controlled by the position of the pulley adjust-

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28. Boyles, Alfred, "<u>The Structure of Cast Iron</u>", American Society for Metals, Cleveland, Ohio, 1947, p. 70.

FIGURE 5. Equipment used in heats N15-N21.
From left to right: fixture for adding magnesium, induction furnace power supply, tube furnace in position over induction furnace, quench tank, and control unit.

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THIS SIDE OF CARD IS FOR ADDRESS

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160 Snyder Hall
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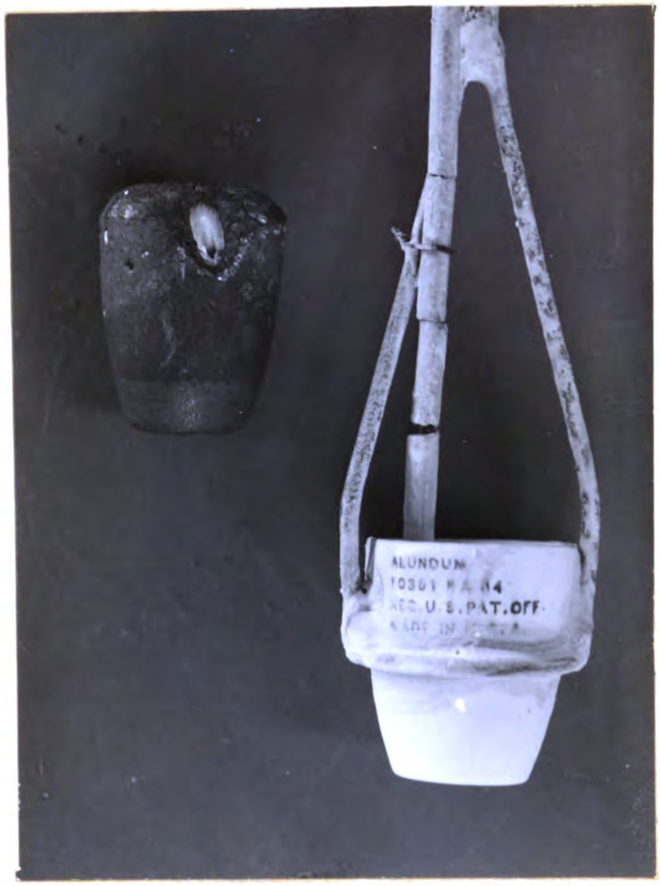

FIGURE 6. Casting and Crucible

ment knob on the potentiometer. Lines on the chart can be easily calibrated to millivolts or directly into degrees F. Figure 7 is a picture of the indicating and recording potentiometer.

After trying this equipment with heat No. N15 it was decided that heat No. N16 would be used to determine the temperature at the beginning and end of solidification. By allowing the sample to solidify completely in the tube furnace, it could also be determined if a sample cooled slowly in the crucible would have a normal structure compared to iron from the same heat cast in a sand mold. A thirty pound heat of the following composition was melted using a 20 K.W. Ajax induction furnace:

Carbon	Silicon	Mn.	S.
3.24%	2.76%	.44	.023

When the temperature reached 2815°F., as measured with a Leads and Northrup optical pyrometer, 0.2% magnesium was added as a 80% nickel 20% magnesium alloy. After the violent reaction of the burning stopped a 0.6% silicon (as 90% ferro-silicon) inoculation was made. The tube furnace was then moved over the induction furnace crucible. The small alundun crucible was lowered down and a sample was taken up into the tube furnace. The temperature of the tube furnace was held constant at 1750°F. The cooling curve of this sample is shown in Figure 10. After the sample was taken the tube furnace was

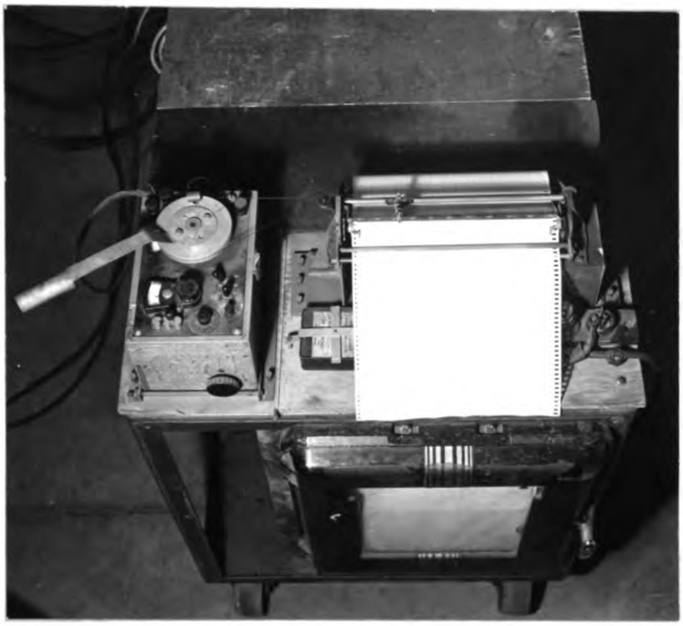

FIGURE 6. Rapid Temperature Recorder

moved away to allow pouring the remainder of the heat. From the cooling curve it was determined that solidification began at 2150°F. and ended at 2080°F. The time required for solidification was four minutes. At the end of nine minutes the sample was lowered from the tube furnace. This was necessary because the temperature of the sample was approaching the temperature of the tube furnace. The point of removal can be seen on the curve.

From heat No. N2 it had been learned that the nodules made their appearance very early during solidification. It was learned from heat No. N16 that solidification of the sample in the tube furnace required four minutes. Using this information it was decided to quench samples at the beginning of solidification, at one minute, at two minutes, at three minutes, and one near the end of solidification.

The sample from heat N15 had already been quenched at the beginning of solidification; N17 was quenched at one minute, N18 after two minutes, N20 after three minutes, and N21 was quenched near the end of solidification.

Cooling curves were plotted for all heats from N15 to N21 and reproductions of these curves are shown in Figures 9 to 14.

Charges for heats N15 to N21 were made up as near alike as possible. Malleable pig iron was used. Composition of the furnace charge was controlled by adding ingot iron and ferro alloys to the charge. The composition of these alloys are shown in Table 1. The amounts used are shown in Table 3. The pig iron was sawed into sections around one inch thick. This was necessary to get the pig into a size which could be charged into the induction furnace. So that the composition could be closely controlled enough pigs were sawed up for seven heats. Pig iron for any one heat came from several different pigs.

The make-up of heats N15 to N21 are shown in Table No. 2.

Table No. 3 is the log of heat N15. In heats N16 to N21 everything was performed exactly the same up to the treatment of the small sample in the tube furnace.

The results of the chemical analysis is shown in Table No. 4. Total carbon was determined by direct combustion in oxygen followed by absorption of the carbon dioxide in soda-asbestos (ascarite). Silicon content was determined by evaporation with perchloric acid. Sulphur was determined by the combustion method. The manganese was determined by the ammonium persulfate oxidation method. Phosphorus could be very closely estimated at .10% from a knowledge of the phosphorus content of the components of the charge.

Table 1. Furnace Charge Compositions (Per Cent)

Malleable Pig		Ferrosilicon		Ingot Iron	
C	4.2	C	0.44	Fe	99.9
Si	1.52	Si	27.4		
S	0.025	S	0.018		
P	0.099	P	0.033		
Mn	0.36	Mn	0.84		

Table 2. Furnace Charge Heat N15 to N21 (pounds)

Charged	
Hanna pig	23.3
Ferro-manganese(80%)	.084
Ferrosilicon(27.4%)	.263
Ingot iron	5.06
Inoculant	
80% nickel 20% magnesium alloy	.3
Ferrosilicon (90%)	.2

Table 3. Log of Heat No. N15

9:45 A.M.	Ingot iron charged on bottom of crucible FeSi and FeMn charged on top of ingot iron Pig iron on top
10:00	Power on 15 K.W.
10:15	Power up to 20 K.W.
11:10	Last of pig added
11:30	Melt down complete
11:45	Temp. 2760°F.
11:50	Temp. 2825°F.
11:51	Pour chill
11:52	Add Magnesium alloy
11:53	Skim and add FeSi inoc.
11:53	Sample taken into tube furnace
11:56	Sample quenched

Table 4. Results of Chemical Analysis

Heat No.	Carbon %	Silicon %	Manganese %	Sulphur %
N2	3.5	1.96	---	---
N15	3.25	2.76	.44	.023
N16	3.24	2.76	.46	.023
N17	3.25	2.76	.46	.022
N18	3.30	2.75	.46	.023
N19	3.29	2.04	---	.025
N20	3.25	2.75	.47	.023
N21	3.29	2.74	.46	.023

Calculated phosphorus Heats N15-N21 0.10%

DISCUSSION OF RESULTS

Samples from heat N2 were quenched at temperatures ranging from molten down to 1000°F. For this heat the equipment shown in Figure 2 was used. Figure 3 is a photomicrograph of a specimen quenched when partially molten. The primary dendrites of austenite are plainly visible. These dendrites have partially transformed into martensite. The intervening spaces between the dendrites show areas of solidification each centered by a graphite nodule. There is strong evidence that the light etching background was molten at the time the specimen was quenched. Figure 4 shows a photomicrograph taken from the sample that was quenched near 1000°F. This sample shows a considerable amount of massive cementite. This cementite was caused by casting the iron in a small section. The size, shape, and distribution of the graphite is very nearly the same as shown in the partially molten sample. The graphite in Figure 3 and Figure 4 is definitely nodular.

Figure 17 is a photomicrograph taken from specimen N15. The cooling curve for specimen N15 is shown in Figure 9. This specimen was quenched at the very start of solidification. The primary dendrites of austenite (transformed to martensite) are easily seen. The matrix is a fine dispersion of austenite and cementite presumed molten at the time of quenching.

Figure 16 is a photomicrograph of a fine shot forced from the partially molten specimen when it was quenched. Graphite was found to be present in these small shot. Positive identification of these small inclusions as graphite was made at high magnification with the use of polarized light. These shot are believed to be of eutectic composition as no primary dendrites were found in them.

Figure 18 is a photomicrograph of specimen N17. The cooling curve for specimen N17 is shown in Figure 11. Specimen N17 was quenched one minute after solidification began. The primary dendrites in this specimen are larger than the ones in N15. This is reasonable as they had more time to form. Specimen N17 shows no graphite in any form.

Figure 19 is a photomicrograph of specimen N18. The cooling curve for N18 is shown in Figure 12. This specimen shows the usual primary dendrites of austenite (now transformed into martensite). Graphite nodules are also visible in the interstices of the dendrites. The austenite cementite background was apparently liquid when the specimen was quenched. The structure of this specimen is very similar to the specimen from N2 shown in Figure 3. It should be noticed in Figure 19 that the structure solidifying around the nodules is different in appearance than the structure of the primary dendrites.

Figure 20 is a photomicrograph of specimen N20. The cooling curve for specimen N20 is shown in Figure 13. This specimen was quenched after three minutes of solidification. Several etching techniques were tried, but the primary dendrites could not be made visible. As can be seen by the photomicrograph very little of the specimen was liquid when the specimen was quenched. The graphite nodule shape and distribution is very similar to sample N18.

Figure 21 is a photomicrograph of specimen N21. The cooling curve for specimen N21 is shown in Figure 14. Specimen N21 was quenched at 3 minutes 45 seconds after solidification began. As can be seen from the photomicrograph very little of the specimen was molten at the time of quenching. In the photomicrograph of specimen N21 the primary dendrites are not visible. There is very little difference in the size, shape, or distribution of the graphite nodules between specimens N18 and N21.

Figure 22 is a photomicrograph of specimenN17K. This photomicrograph was taken of a sample from a keel block casting poured from heat N17. This casting solidified in a core sand mold in a normal manner. The graphite shape and distribution is very similar to specimen N18, Figure 19. Each nodule is surrounded by a ring of ferrite. These ferrite rings were formed after solidification by a secondary precipitation of graphite on the existing nodule. Figure 23 is a photomicrograph from specimen N18K. Sample N18K was taken from a keel block cast from heat N18. This structure is very similar to N17K.

Figure 10 is the cooling curve for specimen N16. In this heat it was established that solidification in this particular set up required four minutes. This heat was also used to demonstrate that a sample of nodular iron cooling slowly in the crucible would solidify in a normal manner. The microstructures of specimen N16 (crucible) and specimen N16K (keel block) were very similar to the photomicrograph of N18K shown in Figure 23.

Early in this work cooling curves were plotted for heavy sections of nodular iron. Comparing these curves to curves of the small sample in the tube furnace it could be seen that the sample in the tube furnace would not cool abnormally slow.

SUMMARY AND CONCLUSION

Figure 3 is a photomicrograph of a specimen that was quenched when partly molten. Primary dendrites are visible and nodules of graphite can be seen in the intervening spaces between the dendrites. In no case can a nodule be seen in or as part of a dendrite. Figure 18 is of a specimen quenched just as the primary dendrites are fully formed. It shows no graphite. Figure 19 is a photomicrograph of a specimen after solidification was half complete. This structure correlates very well with the structure shown in Figure 3.

From information obtained in this work the following conclusions can be drawn.

Solidification in a hypo-eutectic nodular iron begins with formation of primary dendrites of austenite. The graphite nodules then form and nucleolate solidification of the eutectic liquid. The eutectic liquid freezes around these nodules of graphite and primary dendrites. After solidification is complete no new nodules of graphite are formed. However, additional graphite is precipitated on the already existing nodules. This graphite leaving solution causes the familiar ferrite rings surrounding each graphite nodule.

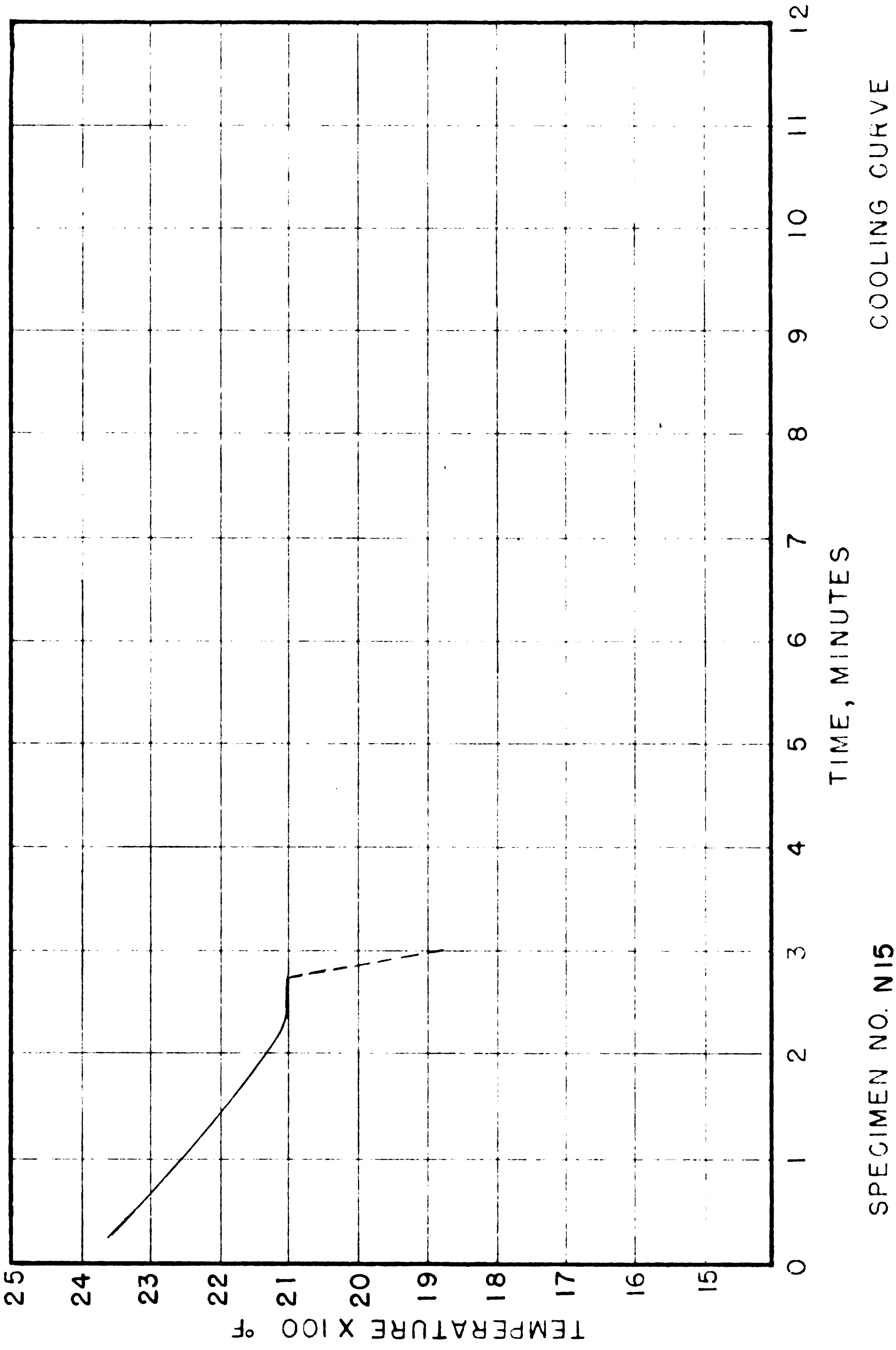
COOLING CURVE
SPECIMEN NO. N15
TIME, MINUTES
TEMPERATURE X 100 °F
0
1
2
3
4
5
6
7
8
9
10
11
12
25
24
23
22
21
20
19
18
17
16
15

FIGURE 9.

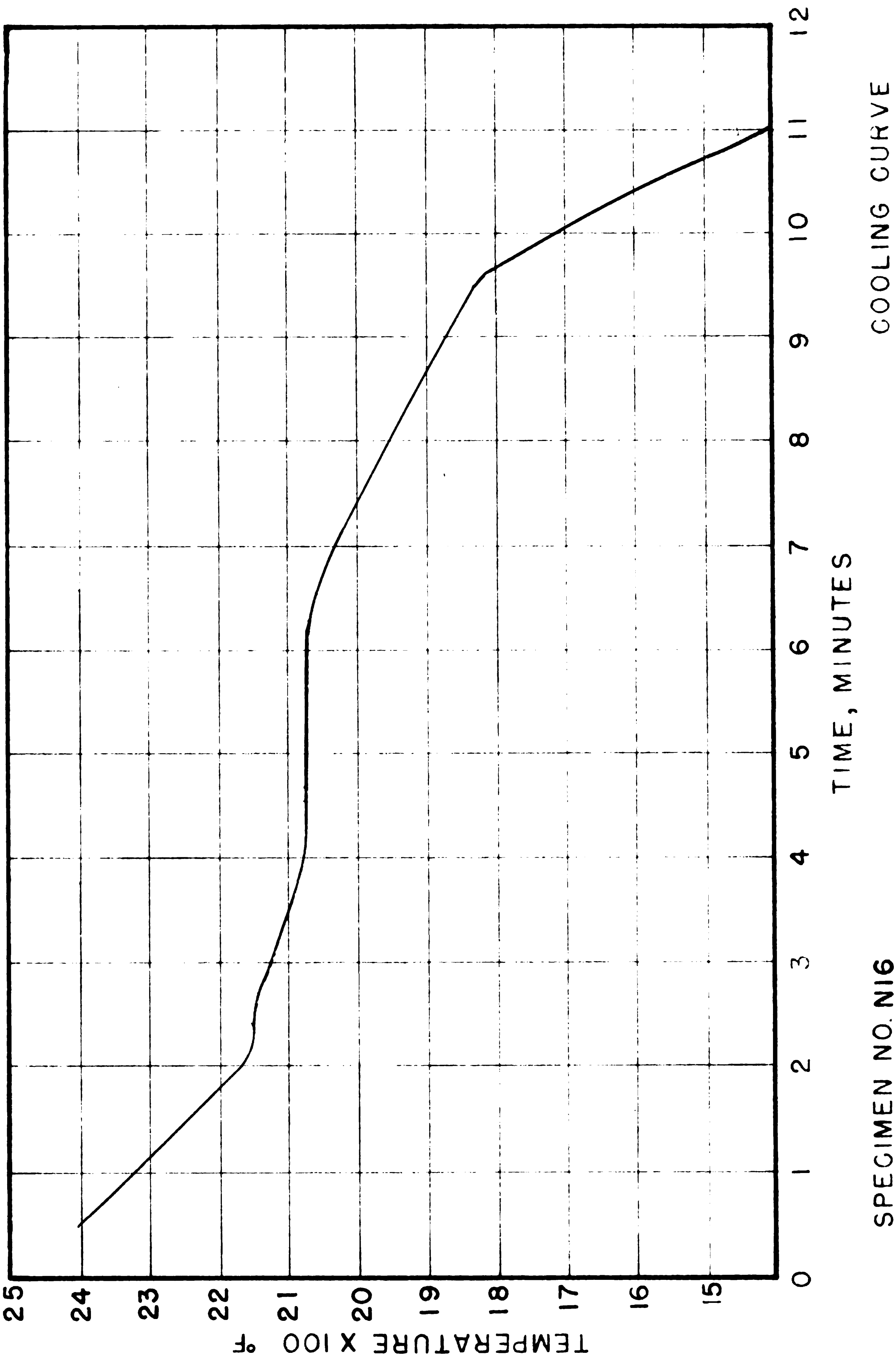
SPECIMEN NO. NI6
COOLING CURVE
TIME, MINUTES
0 1 2 3 4 5 6 7 8 9 10 11 12
TEMPERATURE X 100 °F
15 16 17 18 19 20 21 22 23 24 25

FIGURE 10.

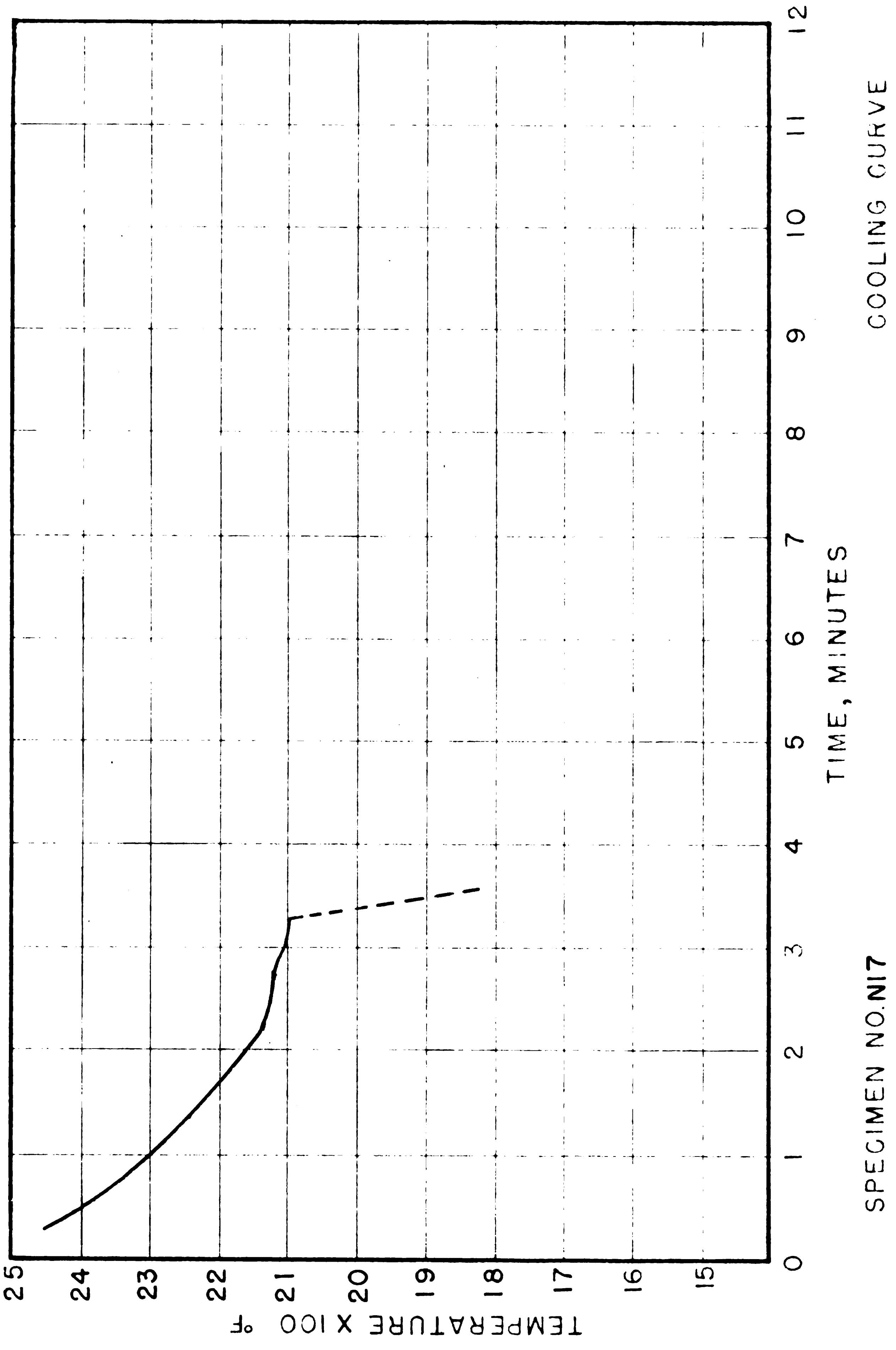
COOLING CURVE
SPECIMEN NO.NI7
TIME, MINUTES
TEMPERATURE X 100 °F
0
1
2
3
4
5
6
7
8
9
10
11
12
25
24
23
22
21
20
19
18
17
16
15

FIGURE 11.

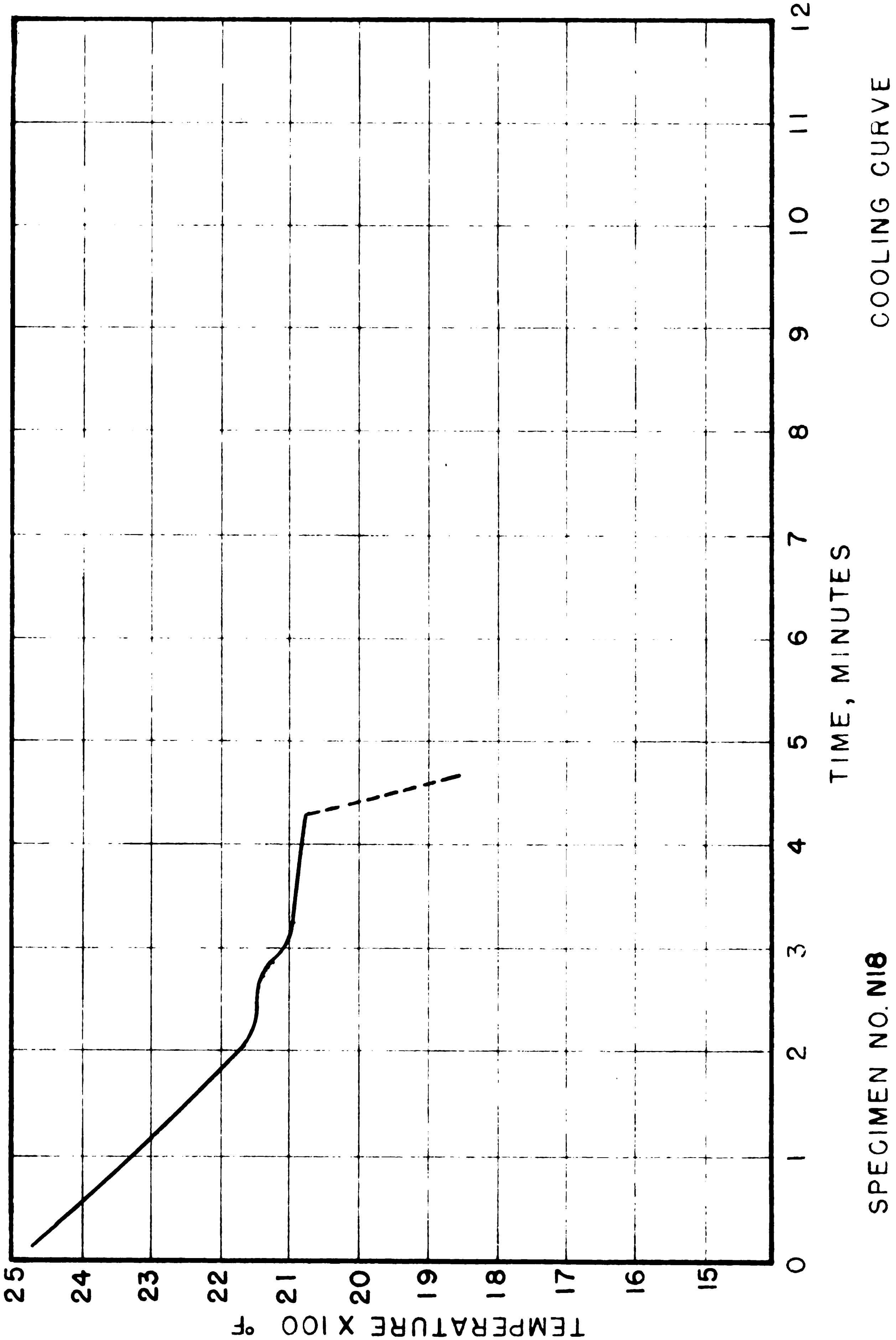
COOLING CURVE
SPECIMEN NO. N18
TIME, MINUTES
TEMPERATURE X 100 °F
0 1 2 3 4 5 6 7 8 9 10 11 12
25 24 23 22 21 20 19 18 17 16 15

FIGURE 12.

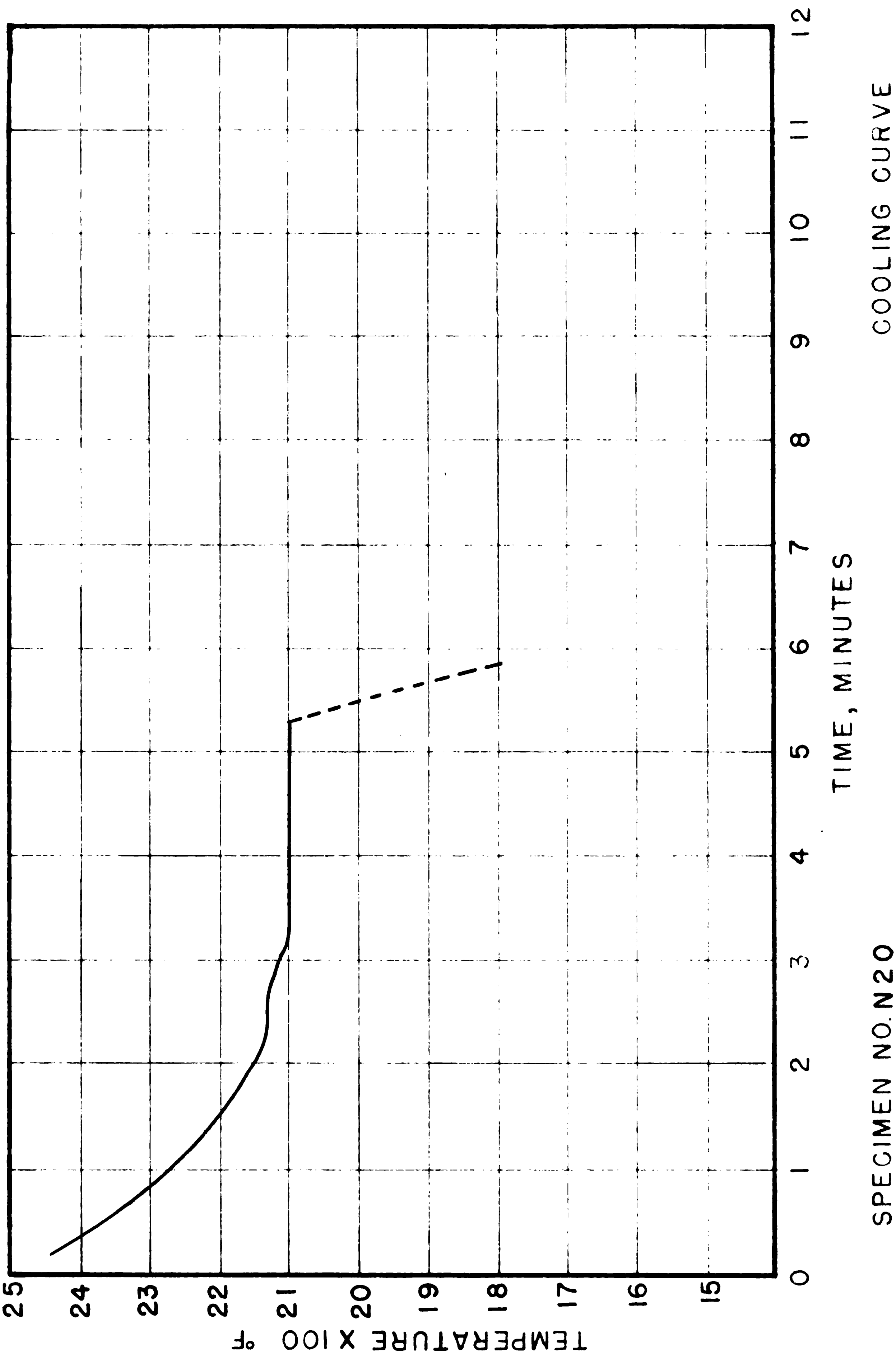
COOLING CURVE
SPECIMEN NO. N20
TIME, MINUTES
0
1
2
3
4
5
6
7
8
9
10
11
12
TEMPERATURE X 100 °F
25
24
23
22
21
20
19
18
17
16
15

FIGURE 13.

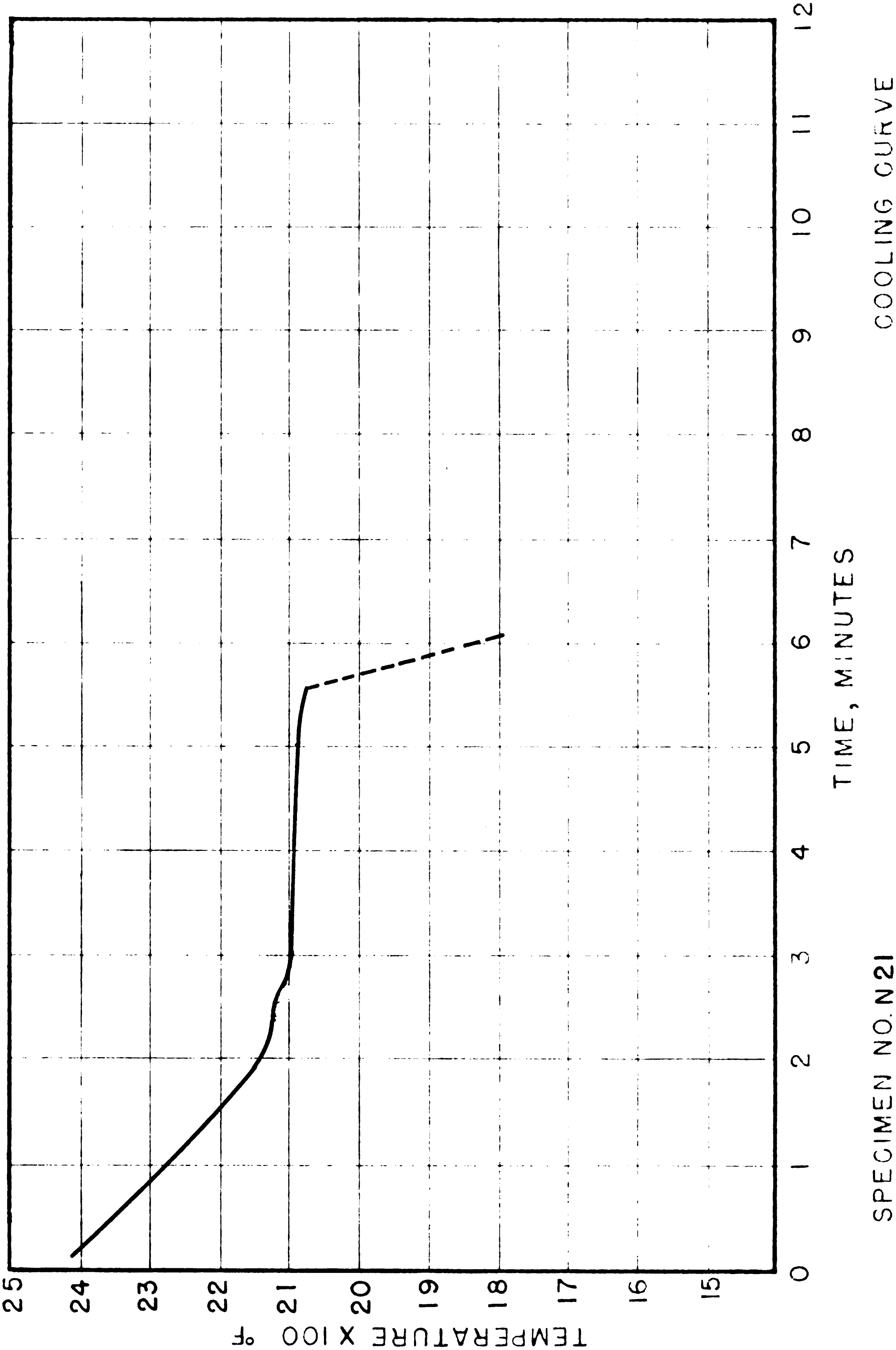
COOLING CURVE
SPECIMEN NO. N21
TIME, MINUTES
TEMPERATURE X 100 °F
0 1 2 3 4 5 6 7 8 9 10 11 12
25 24 23 22 21 20 19 18 17 16 15

FIGURE 14.

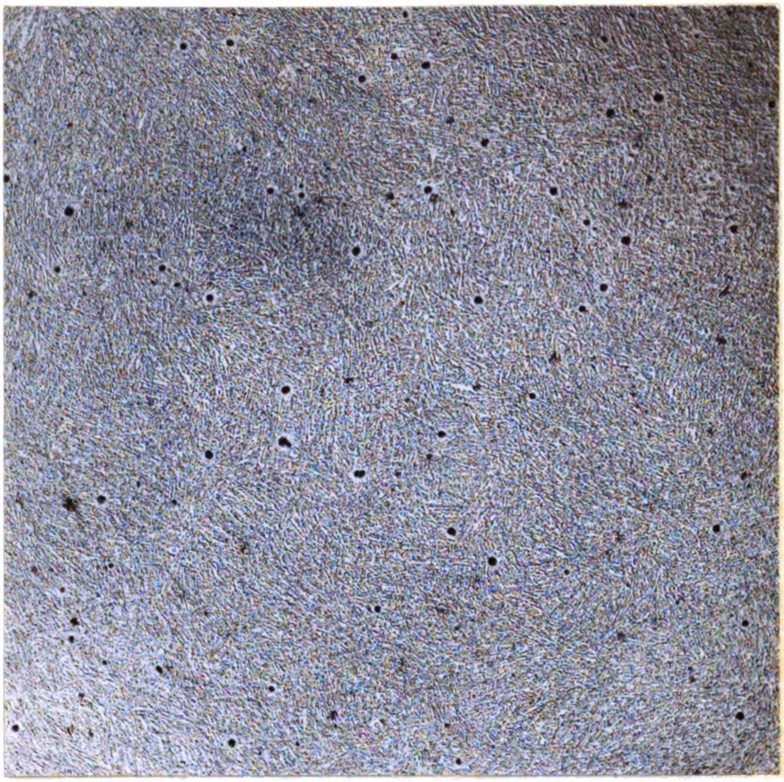

FIGURE 16. Heat No.N15.

From small shot forced from specimen

No. N15. Magnification: 75 X

FIGURE 17. Heat No. N15
From specimen quenched at the
beginning of solidification.
Magnification 75X

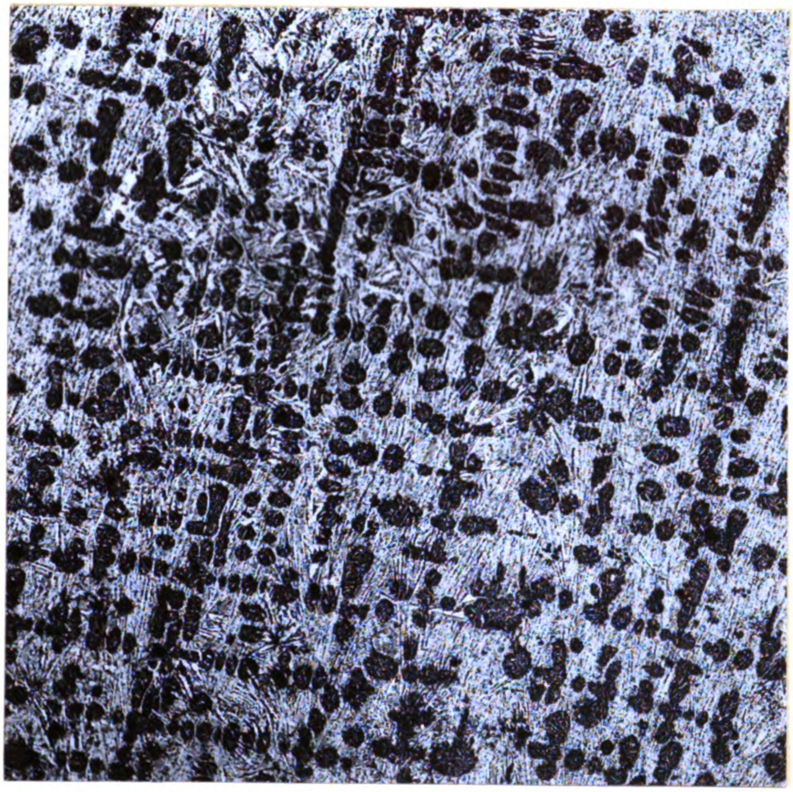

FIGURE 18. Heat No. N17
From specimen quenched one minute
after beginning of solidification.
Magnification 75 X

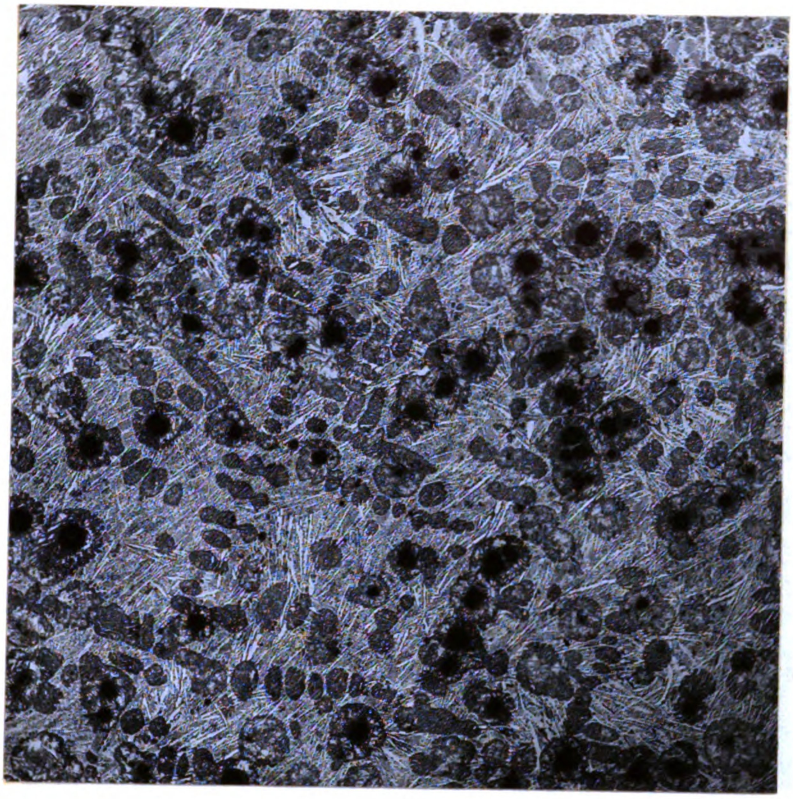

FIGURE 19. Heat No. N18

From specimen quenched two minutes after beginning of solidification.

Magnification: 75 X

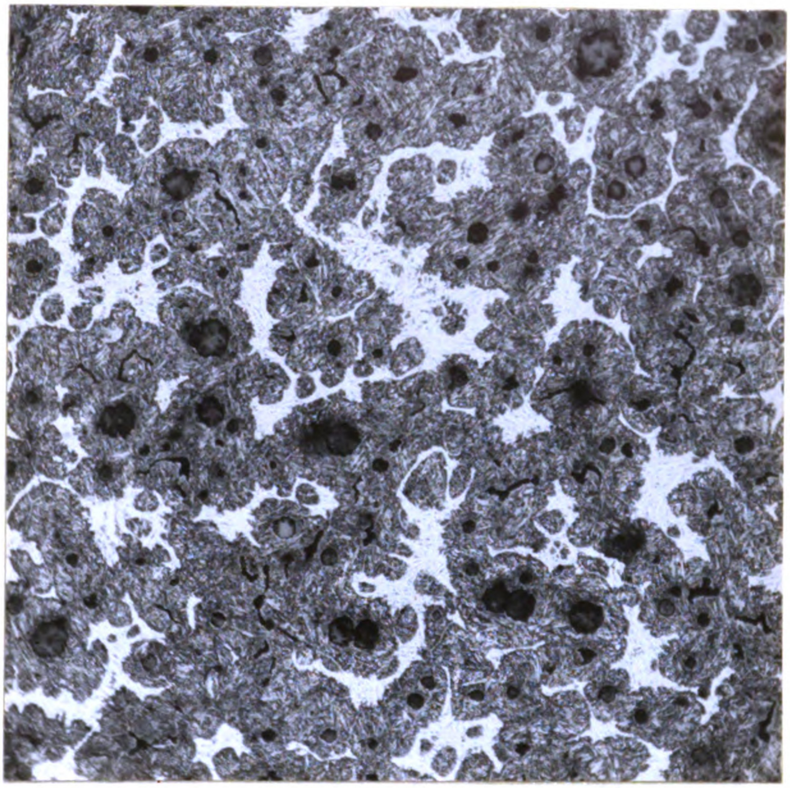

FIGURE 20. Heat No. N20.
From specimen quenched three minutes after beginning of solidification.
Magnification: 75 X

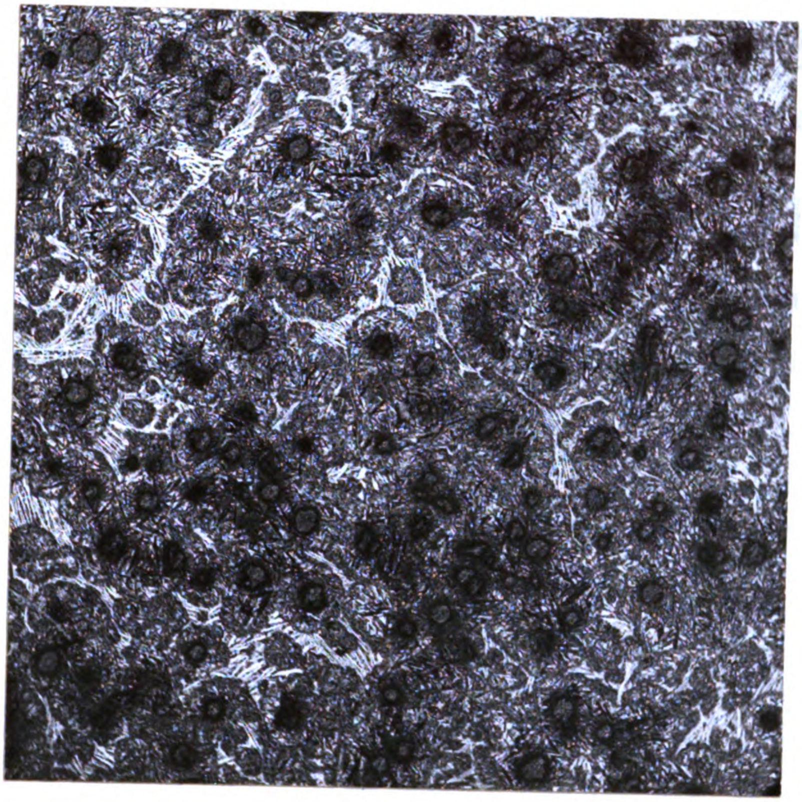

FIGURE 21. Heat No. N21.
From specimen quenched near the end of solidification.
Magnification 75 X

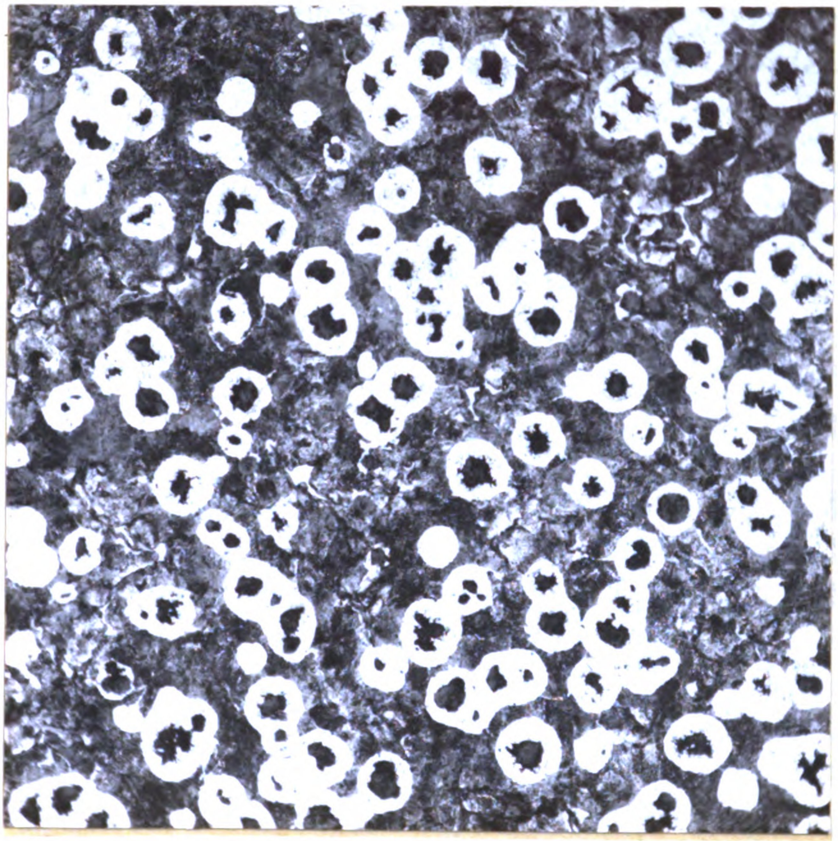

FIGURE 22. Keel Block

From heat No. N17, specimen N17K.

Magnification: 75 X

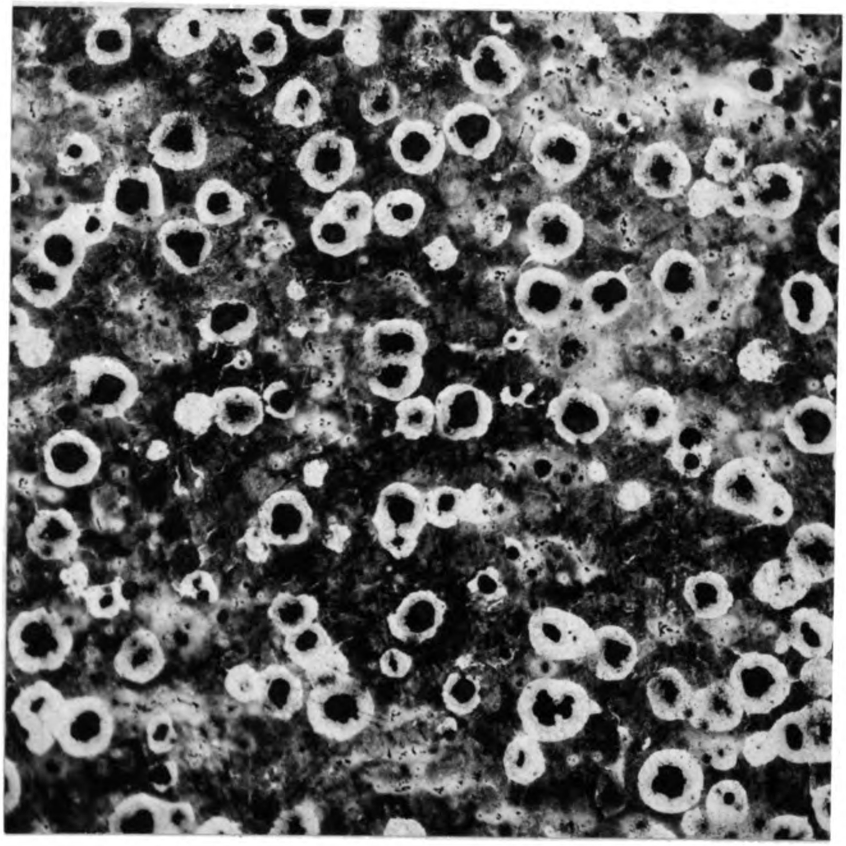

FIGURE 23. Keel Block

From heat No. N17, specimen N17K.

Magnification: 75 X

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