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THESIS

Steel

Title: Grain
formation in steel

Chemical engineering

Motion Picture Studies
of
The Grain Formation
In Steel
At Elevated Temperature
and
Under Reduced Pressure

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PLASIS

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TABLE OF CONTENTS

Introduction -----	Page 1
Apparatus -----	Page 5
Procedure -----	Page 11
Experimental -----	Page 12
Discussion -----	Page 24
Summary -----	Page 29
Bibliography -----	Page 30
Film Index -----	Page 31

INTRODUCTION

Many very important changes take place both in the physical properties and in the microstructure in steel at temperatures exceeding that of room temperature. The changes in the physical properties such as the yield point, tensile strength, elongation, etc., have been studied extensively by many investigators. In the majority of cases test bars have been subjected to tensile tests while maintained at some predetermined temperature.

The changes in the microstructure, or the allotropic transformations in low, medium, and high carbon steels and in a variety of alloy steels, have been studied by a great number of workers. There is yet no theory or explanation of these allotropic forms or transformations that is universally accepted by all. A comparatively few have studied the transformations as they actually take place, that is, by observing the transformations through a microscope as it occurs in the metal. The changes in the microstructure have been studied by heating steel in a vacuum or supposedly neutral atmosphere of hydrogen or nitrogen, and the resulting structure assumed to be that which existed at the highest temperature reached. Other investigators have heated polished specimens in a vacuum and then admitted some etching gas (chlorine) allowing it to remain for a few moments and then pumping it out. The specimens were then cooled in the furnace and the structure assumed to be that which existed at the temperature the etching gas was admitted into the furnace. Others heated polished specimens in a vacuum while under microscopic

observation and interpreted the surface changes which took place as those brought about by a change in volume or by allotropic transformation.

B. A. Roger (7), by means of a motion picture film showed the A_3 transformation in Armco iron as an eruptive wave which appeared at one side of the field of view and swept over the surface. The time of passage of such a wave being a fraction of a second or several seconds, depending upon the rate of heating.

H. J. Wiester (9), at Technischen Hochschule, Berlin, succeeded in watching the crystallization of martensite directly in the microscope and in photographing the transformation on a motion picture film.

Wiester obtained austenite by quenching steel with 1.7 percent carbon in a metal bath at 100 C. The austenite is extremely stable at this temperature, and when the steel was allowed to cool, martensite crystallized out from the austenite grains. The changes involved an increase in volume, and was the result of two opposing tendencies; the immobility of the carbon atoms which obstruct atomic rearrangement, and the tendency of the iron atoms of the undercooled austenite to expand into the alpha space lattice.

T. D. Parker (3) designed a small vacuum electric furnace to set on the stage of a microscope. The furnace was so constructed that he could observe, through a small glass window in the top a specimen as it was heated in a vacuum through the critical range.

Parker used normalized specimens of low and medium carbon steel. He observed several interesting facts, among which was the volatilization of elements from the polished surface, or decarburization, and the volume changes at temperatures within the critical range. He did not observe any dissolving reaction of alpha and gamma iron or the A_3 transformation as an eruptive wave passing over the surface.

K. L. Clark (3) followed the same method of investigation, with a few modifications, as did Parker. In his investigation hardened high carbon steel was used, and when this steel was heated in a vacuum, a heat-etched pattern appeared in a similar manner as observed by Parker. Upon the normalized steel the characteristic cementite net-work was formed and upon the quenched steel a mass of acicular needles. Clark did not observe a wave pass over the surface of the metal as it passed through the A_3 transformation range.

Rawdon and Scott (6), in their work examined the structure of iron and mild carbon steel at high temperatures by heating polished specimens in a vacuum. They interpreted the heat-etched pattern produced on the polished face as the structure corresponding to the different allotropic forms. They also state that the pattern produced by heating revealed not only the condition of the surface metal, but also that of the interior.

The present investigation was performed in a similar manner as

followed by Parker and Clark, i.e., heating a specimen in a vacuum while under observation. Motion picture apparatus was used to record the changes in the structure which took place on the polished surface of the specimen.

APPARATUS

The apparatus used in this work consisted mainly of a small vacuum electric furnace, microscope, Cine'-Kodak motion picture machine, model A, and illumination system (Figure 1).

The furnace (Figure 2) was the one used by Parker in his work. The method of supporting the specimen was changed (Figure 3) so that it would not be necessary to remove the heating element each time a new specimen was used. The furnace was designed so that it could be placed on the stage of a Bausch and Lomb microscope (Figure 3a). Holes drilled in the base of the furnace provided for the entrance of the thermocouple and heating element leads. A horizontal hole was drilled through the base and connected to a water line for cooling purposes. A silica ring supported by a small flange on the furnace base held a small steel cylinder which had a one-fourth to one-eighth inch tapered hole in the center. The specimen was machined to fit this tapered hole, thus giving it a fairly rigid support. Supporting the specimen in this manner facilitated the removal and insertion of a new sample. Temperature measurements were made by a Leeds and Northrup potentiometer using an iron-constantine thermocouple made of B. and S. gauge 25 wire. The thermocouple passed through the silica ring and steel cylinder into a one-eighth inch hole drilled in the steel specimen. The end of the couple was one-eighth inch or less below the polished surface of the specimen and the temperature recorded by the couple was assumed to be that of the polished face.

Figure 1
Apparatus

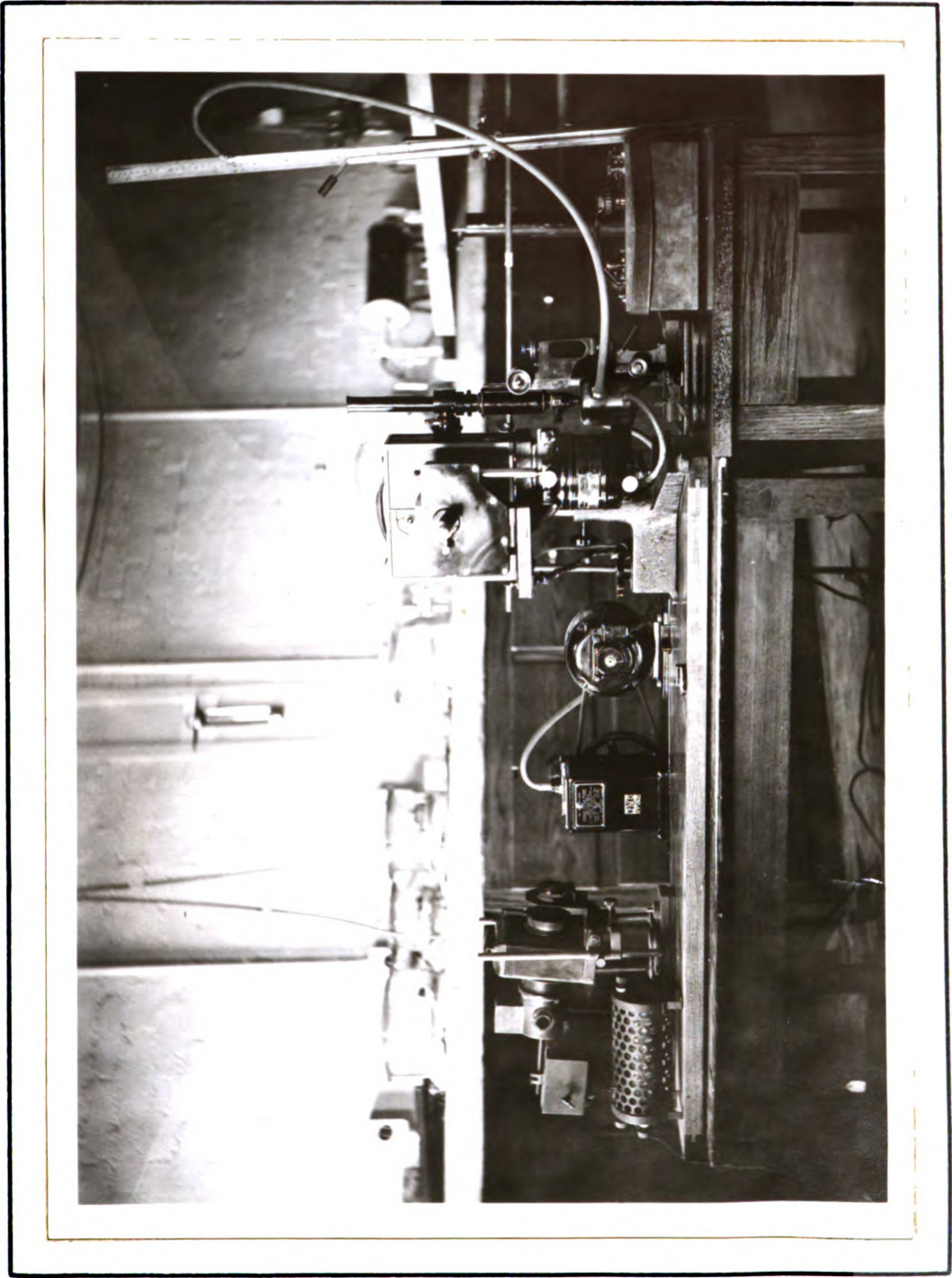




Figure 8
Image designed
and
used by Parker.
Picture by Parker.

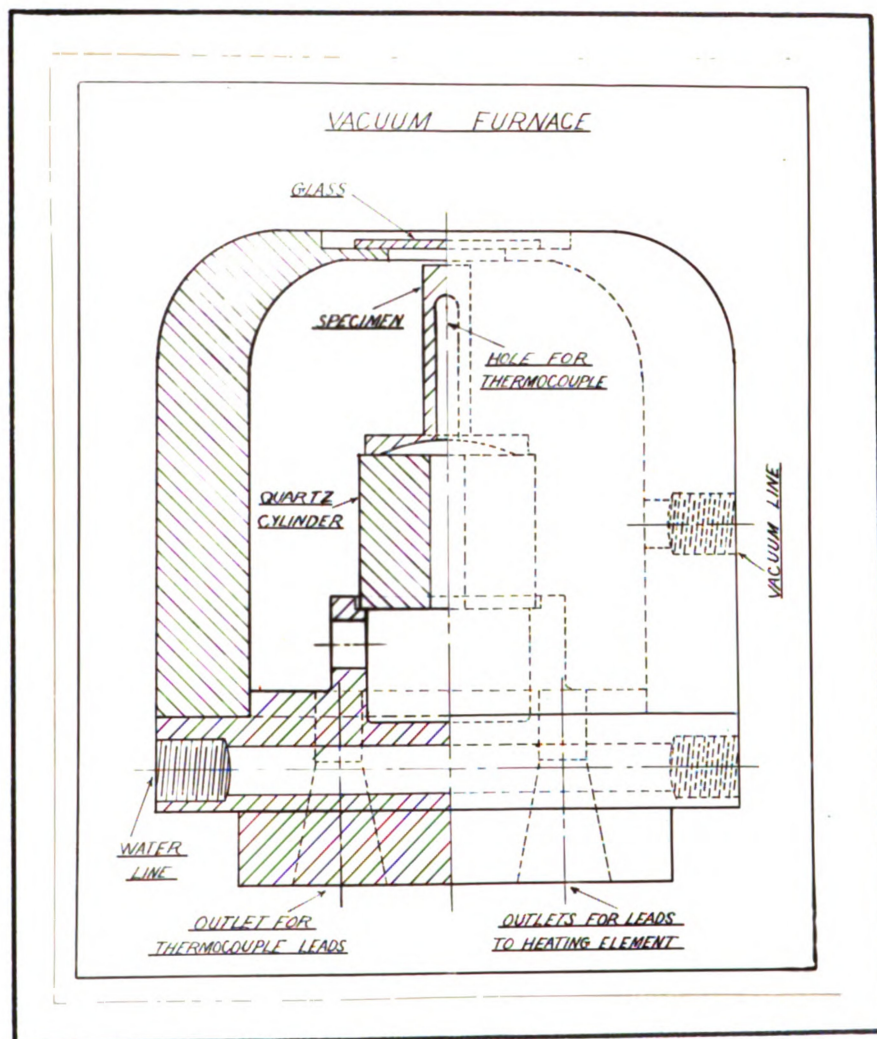


Figure 2
A component of γ of an
ad part.

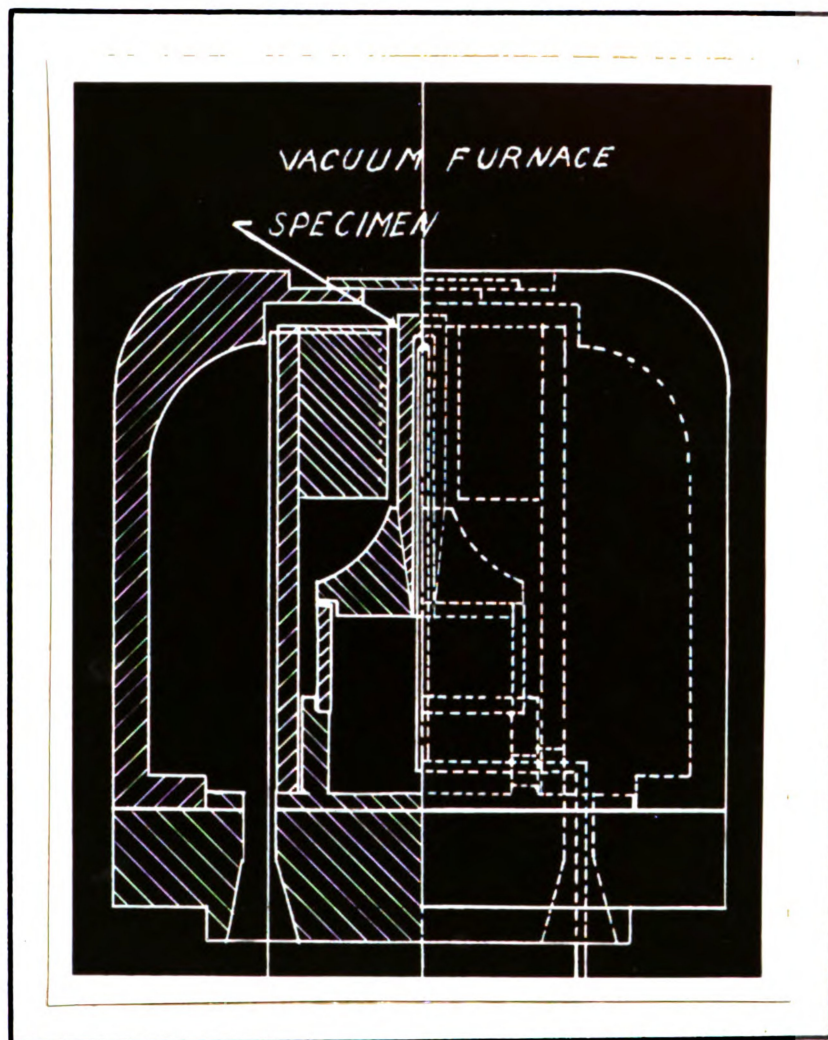
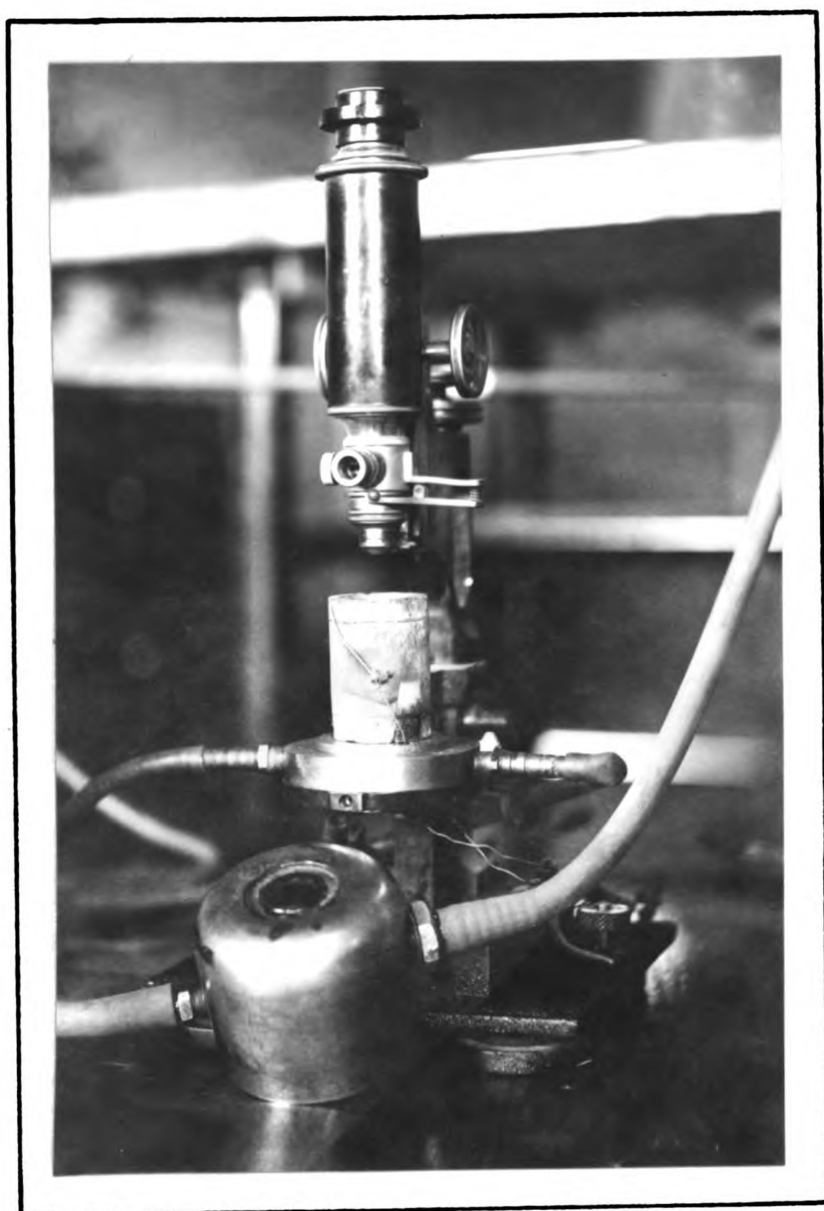


Figure 1a
Furnace with out top
and
Microscope.

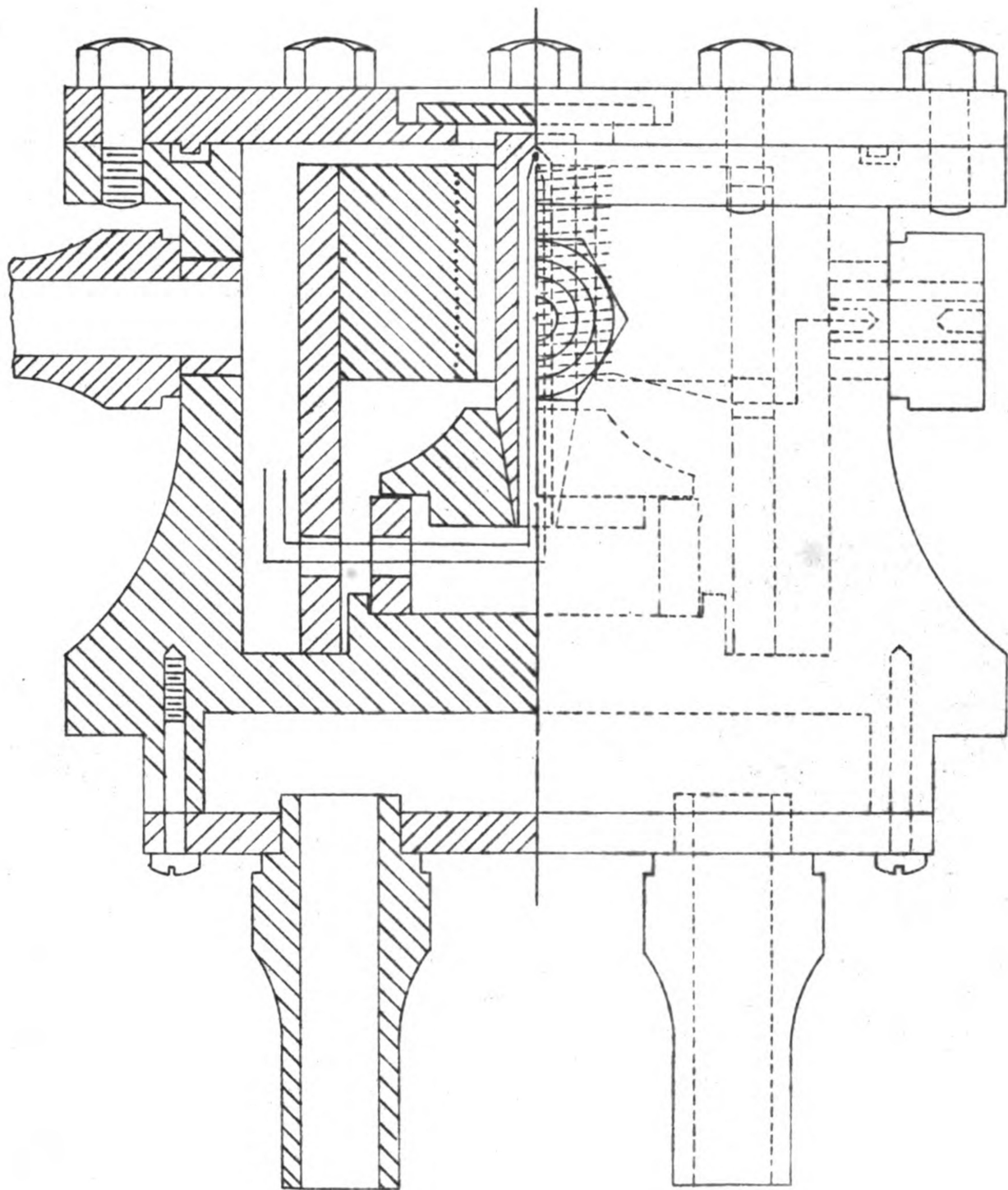


The top of the furnace was equipped with a glass window which made the specimen visible from the outside. The height of the furnace was such that the distance from the top of the specimen was less than the working distance of a 16 mm. objective. A gravity seal was maintained between the base and the top of the furnace. The contact surfaces were ground together with different grades of abrasives to give a true plane surface. It was necessary to pass a stream of air over the glass window to keep it cool when the furnace was being heated.

It was difficult to maintain a vacuum in the furnace as designed by Parker. The contact surface between the top and bottom would leak, unless perfectly true, and had to be frequently reground. The method of inserting the heating element and thermocouple leads was very unsatisfactory, since they were passed through rubber stoppers cemented in the base of the furnace and covered with De-Khotinski cement. Thus, whenever the lead wires were moved, the cement was apt to be cracked and a vacuum could not be maintained.

In order to overcome these difficulties, a new furnace was designed and constructed (Figure 4). The details of construction are shown in the accompanying blue prints. The furnace was made much lighter and smaller, being only three inches in diameter and about two and one-half inches high. The top of the furnace was made in the form of a flat plate with a small flange on the under side. This flange was machined to fit into a small groove containing a lead gasket, in the body of the furnace. The top was secured to the

Figure 4
New furnace design



P. W. 9.201

body by six machine screws. This method gave two contact surfaces and also the additional advantage of the lead gasket.

The furnace was so designed that the heating element and thermocouple lead-ins are permanently sealed into the wall of the furnace. A test sample may be removed or replaced without altering or touching the electrical or thermocouple leads on the inside of the furnace. The blue print No. 5 shows the details of assembly. The external connections to the furnace are made by plugging into the permanently embedded brass lead-ins.

The window in the top had to be changed from glass to fused silica to withstand the temperature change and prevent breaking. Transparent Vitreosil or fused silica discs were used which had been ground and polished. (Discs obtained from the Thermal Syndicate Ltd. 58 Schenectady Ave., Brooklyn, N. Y.)

Other details of the furnace, i.e., the heating element, method of supporting the specimen and water cooling the base, etc., were not altered.

The electric heating element, made of chromel resistance wire, (B. & S. gage 18) was wound in a spiral. The spiral was about one-fourth inch larger in diameter than the shank of the specimen. It was cemented, with alundum refractory cement, into a silica tube and placed around the shank of the specimen and supported on the base of the furnace as shown in Figure 3.

The microscope used was of the type generally employed in metallography. (Manufactured by Bausch and Lomb.)

The apparatus for taking the motion pictures consisted of a Cine'-Kodak Model A without lens, support for Cine'-Kodak with motor, speed control, place for supporting microscope, optical connector, and observation eyepiece. (1)

The support of the Cine-Kodak has a 110 volt 60 cycle synchronous motor mounted on it, which, through a chain of gears, drives the camera at one of eight different speeds. The gears are inclosed in a sound proof box and the functioning of the gears and the running of the motor induces but a negligible amount of vibration, which is a very important factor. The speeds are controlled by an external gear shift drive having eight rings numbered from one to eight. The slowest speed permits pictures to be taken at the rate of one every four minutes and sixteen seconds, while the highest speed is sixteen pictures every second. The following table gives the different speeds for the different gear positions.

Gear Position	Picture Speed
1	16 per second
2	8 per second
3	2 per second
4	1 every 2 seconds
5	1 every 8 seconds
6	1 every 32 seconds
7	1 every 2 minutes and 8 seconds
8	1 every 4 minutes and 16 seconds

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The support carrying the camera, motor, and gear train is adjustable for height. Into the camera is fastened an optical connector and observation eyepiece. The microscope sets on a base plate so that the body tube and eyepiece adapter are directly under the connector. Into the eyepiece adapter of the microscope is inserted an amplifier, either a 7.5 X, 12.5 X, or 15 X, depending on the magnification required. The observation eyepiece is equipped with a system of prisims so that observation and focusing adjustments can be made while the camera is in operation.

The electric motor is operated by 60 cycle current, the alterations of which are constant. With the shift lever set at a certain number of exposures per minute, or per second, there is very little deviation from this speed. The importance of this fact lies in the advantage of being able to take a film when it is developed, and by counting the number of frames during which a complete process or change has taken place, determine the exact rate at which it took place.

The following pieces of apparatus were also used:

Cenco Hyvac Pump

Mercury manometer

Leeds and Northrup potentiometer

Carbon arc lamp

Variable resistance

Electric heating element

Steel specimen

Air line

Water connections

Mazda ribbon filament lamp

A. C. 110-volt, 60 cycle current

D. C. 220-volt line, used for heating

PROCEDURE

The procedure for a characteristic experiment can be described briefly in the following manner. In all experiments the specimens were of 1020, 1040, or 1050 steel. The samples were first heated in a vacuum bomb at 1800 F for a period of three hours so as to produce a large grain structure and to remove the occluded gases. They were then machined down to size. (One-fourth inch in diameter and one and one-quarter inches in length.) The specimens were prepared for metallographic examination by the usual procedure. The sample was then placed in the furnace. The contact surfaces of the top and base were wiped clean and then a thin film of vaseline was applied and the top placed in position. The vacuum pump was started and allowed to run for twenty minutes or more before the furnace was heated. The cooling water was adjusted and camera placed in position. When the specimen had reached a temperature of between 500° F and 800° F the microscope was focused and the camera started. As the temperature increased the microscope had to be refocused due to the expansion of the specimen. During the experiment the temperature was recorded each minute, and during several runs, for each foot of film used. From 25 to 35 minutes were required for the furnace to reach maximum temperature. The specimen was allowed to cool in the furnace so that the surface would not become oxidized and it could be examined when removed.

EXPERIMENTAL

In this work a new method of recording the changes which take place on the surface of heated steel was employed. Instead of the customary still pictures, a motion picture camera was used to obtain moving pictures. There was no literature or information available concerning the time of exposure or the speed at which the camera should be run for this method of operation. Thus considerable time and experimentation were required to obtain a satisfactory combination of light intensity and shutter speed.

A short exposure of film was made for each of the eight different shutter speeds. A carbon arc lamp was used as the source of light. The results of this first film showed the sixth speed (one exposure every 32 seconds) gave the right time for exposure, but as the change took place rapidly, this speed was too slow to record the details. The preceding speeds did not give sufficient time for exposure, thus causing the film to be light. Speeds seven and eight were slow, causing the film to be over-exposed and dark.

The camera is equipped with a small disk which has in it four smoked glasses of different densities. The smoked glasses are marked 0.0, 0.3, 0.6, 0.9, and henceforth will be referred to as 0.3 smoked glass, etc. The following table shows the amount of light which passes through each glass:

Smoked glass	Percent of light
0.0	100%
0.3	70%
0.6	40%
0.9	10%

A sample of film was made with the camera set as Number six speed and using each of the smoked glasses. When this film was examined it was too dark, being over-exposed. A similar sample of film was made for speeds five, four, three and two, using each of the smoked glasses at each different speed. The portion of the film obtained using Number four speed and the 0.6 smoked glass gave about the right exposure. Also, Number three speed with the 0.0 and 0.3 smoked glass could be used, but it was too fast. The time of exposure being one-half second, giving 120 pictures or frames per minute and requiring three feet of film. Use of this speed would require a great amount of film, involving unnecessary expense.

The lighting arrangement was changed to see what effect a less intense source of light would have on the shutter speed. The carbon arc lamp was replaced by a mazda ribbon filament lamp, and a film made with this arrangement. The camera was run at number seven speed and a short exposure made using each smoked glass. Upon developing the film it was found to be over-exposed. Slight detail could be seen on the portion made when the 0.9 smoked glass was used, but not for the others. The same procedure was followed for speeds four, five, and six. The developed film showed that speed five with 0.9 and 0.6 smoked glass gave about the right exposure. Also speed four with the 0.0 and 0.3 smoked glass.

A film was made using an unetched sample of 1020 steel. The camera was run at Number four speed (one exposure every two seconds)

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and the 0.3 smoked glass was used to filter out some of the light. During the heating the temperature was recorded each minute, and at the end of each foot of film used. From this data a heating curve was plotted. (Number 1) From this curve it was possible to determine the temperature of the specimen at which each individual frame was exposed. This is possible due to the fact that the camera runs at a constant rate of speed. This film did not show grain formation very well because the specimen was slightly oxidized, obliterating the heat-etch

Data for Curve No. I

Time	Temperature	Feet of Film	Temperature
1	200° F	0	650° F
2	310	1	685
3	400	2	770
4	460	3	860
5	515	4	910
6	570	5	960
7	---	6	1000
8	650	7	1015
9	690	8	1035
10	770	9	1100
11	845	10	1170
12	885	11	1215
13	930	12	1230

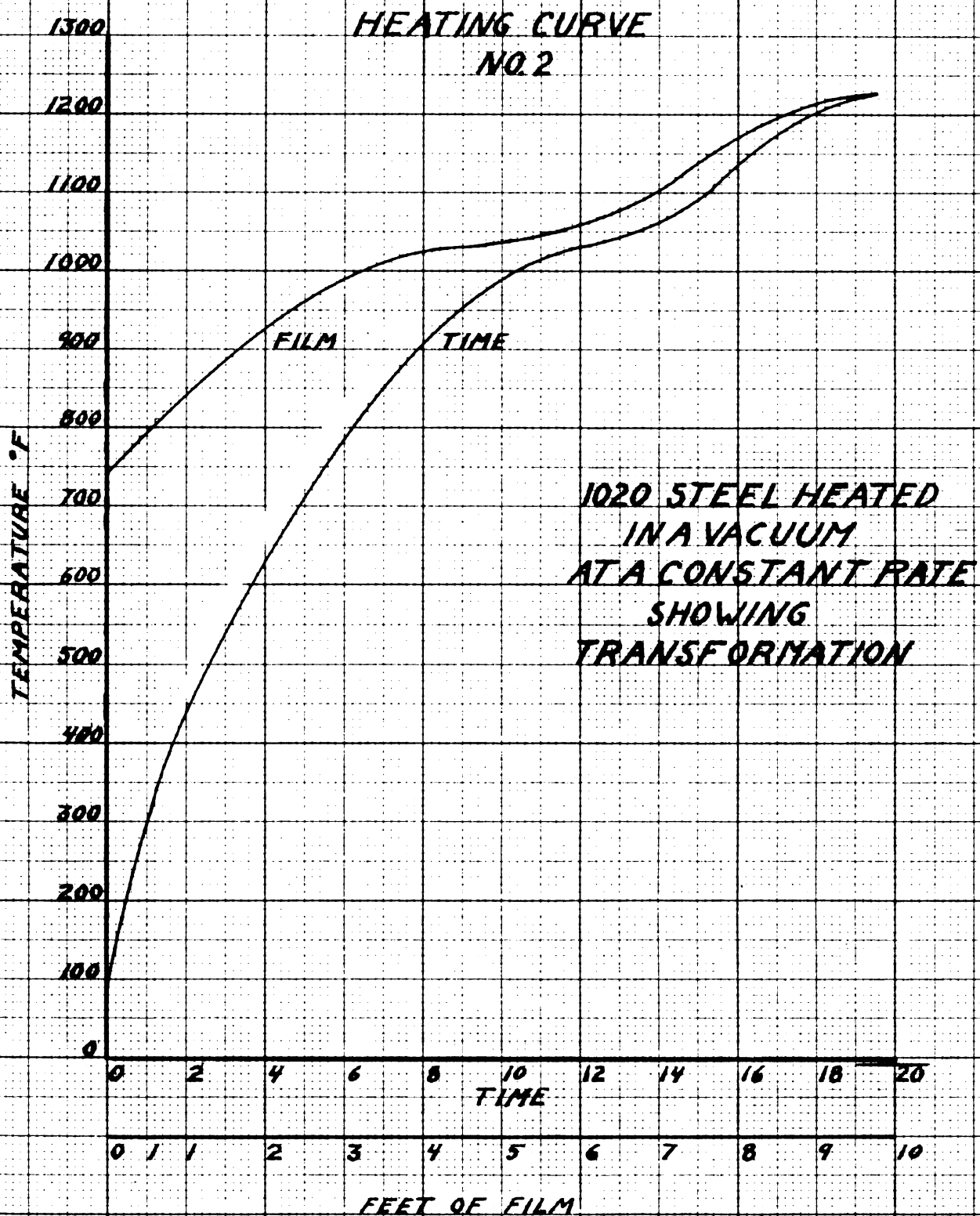
Time	Temperature	Feet of Film	Temperature
14	960	13	1260
15	990	14	1300
16	1010		
17	1060		
18	1100		
19	1160		
20	1205		
21	1230		
22	1250		
23	1290		
24	1315		
25	1330		

A short film was made using an unetched sample of low carbon steel (1020). The camera was run at number four speed and the 0.3 smoked glass used, the same as in the preceding trial. The film obtained was not clear due to vibration. If the microscope and adapter were not placed exactly under the observation eyepiece or if the observation eyepiece was touching it, the vibration of the motor was imparted to the sample, causing the film to appear blurred. The following is the time temperature data recorded for the trial and the data for plotting heating curve Number 2:

Heating Curve No. 2

Time	Temperature	Feet of Film	Temperature
0	72°F	0	750°F
1	260	1	860

Time	Temperature	Feet of Film	Temperature
2	480°F	2	955°F
3	580	3	995
4	650	4	1030
5	750	5	1050
6	800	6	1070
7	860	7	1120
8	900	8	1185
9	940	9	1205
10	995	10	1270
11	1025	11	1310
12	---		
13	1040		
14	1060		
15	1075		
16	1135		
17	1180		
18	1200		
19	1220		
20	1250		
21	1300		
22	1340		



The two heating curves show that there was some change taking place between 1000° F and 1200° F. It was impossible to observe any detail on the film. During the run the microscope had to be readjusted several times due to the expansion of the specimen. Very often this resulted in bringing a different field under observation that was originally observed. Thus, the film recorded a short length of one field, and then another, and not a continuous picture of the same field as desired.

Figure 5, taken at 100 X shows a sample of low carbon steel after it was cooled from 1300° F. The grain structure is much smaller than in the original nital etched specimen. The pearlitic areas have completely disappeared. The grain boundaries are much larger than in a specimen etched with acid. Figure 6 shows the same specimen at 500 X. There appears to be small globules forms in the grain boundaries.

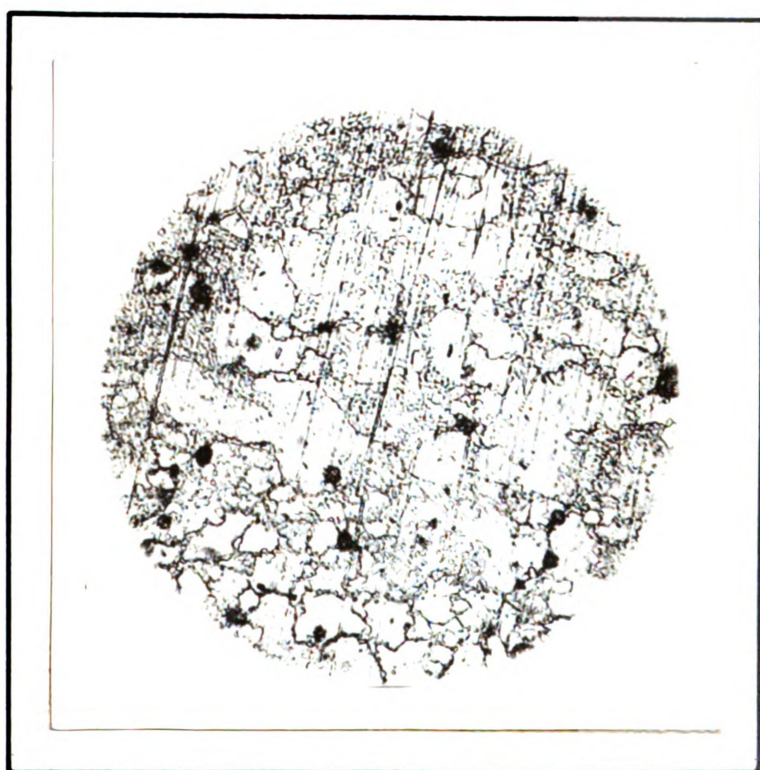
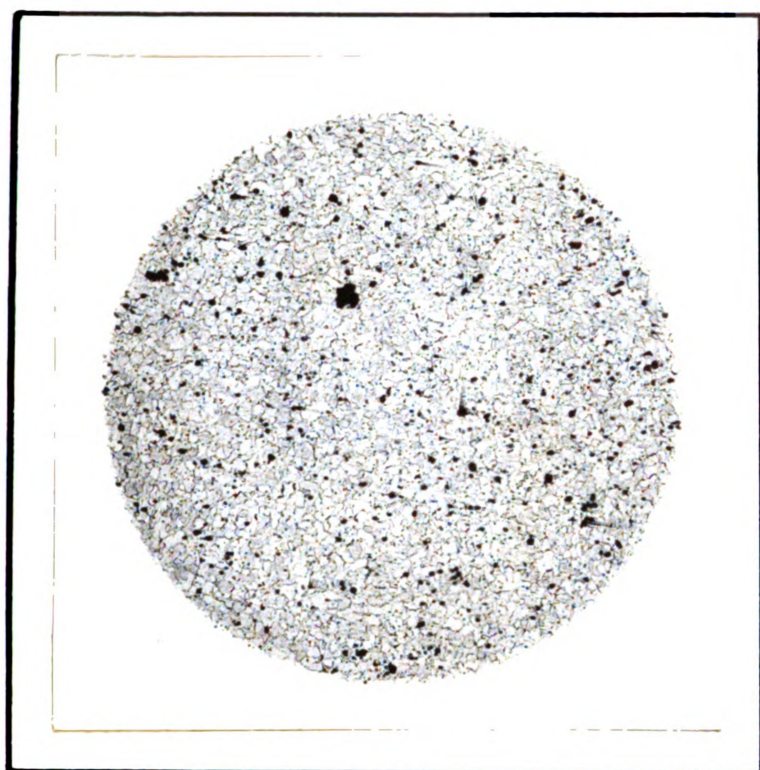
The motion picture of this specimen showed a formation very similar to that shown in Figure 6. The first appearance of a heat etch was at about 1000° F.

There was no heat-etched pattern on the specimen when the film was started (800° F), but as the temperature increased, a faint pattern appeared, and as the temperature continued to rise it became more and more distinct until about 1250° F when there seemed to be no further change.

A sample of 0.4 carbon steel was placed in the furnace unetched

— Figure 5
Photomicrograph 100 X
Heat etched. Cooled from
1350° F

Figure 6
Photomicrograph 500 X
Same as above.



and heated to 1400°F. The film obtained shows the heat-etch or network as it first starts to form between 1000°F and 1250°. At about 1350°F the microscope had to be refocused and a different field was obtained which did not appear to be the same as the original field.

Figure 7 shows the structure at 100 X of a specimen after cooling from 1500°F. It appears quite rough and the boundaries are not distinct. The pearlitic areas have completely disappeared. In Figure 8, photomicrograph taken at 500 X, there is some evidence of a second net-work or set of grain boundaries. Figure 9 taken at 1000 X of the same area shows more clearly the roughened condition of the surface, and also the widening of the grain boundaries. There are also small globules formed, some in the grain boundaries, and some in the interior of the grains. These may be spheroids of cementite or may be caused by some surface phenomena. The film obtained during this trial shows the formation of the heat-etch or the recrystallization, but it did not have very good detail. The film was started at 850°F and was continuous until 1500°F was reached, after which temperature there seemed to be no further change.

A double net-work or two sets of grain boundaries are shown in Figure 12, a photomicrograph taken at 1000 X. The focal points of the two net-works are different. When the specimen was at the elevated temperature there was just one net-work or set of grain boundaries and this appeared to be the one consisting of the wide grain boundaries. This indicated that one set was formed as the specimen

Figure 7

Photomicrograph 100 X

Heat etched. Cooled from

1500° F

Figure 8

Photomicrograph 500 X

Same as above.

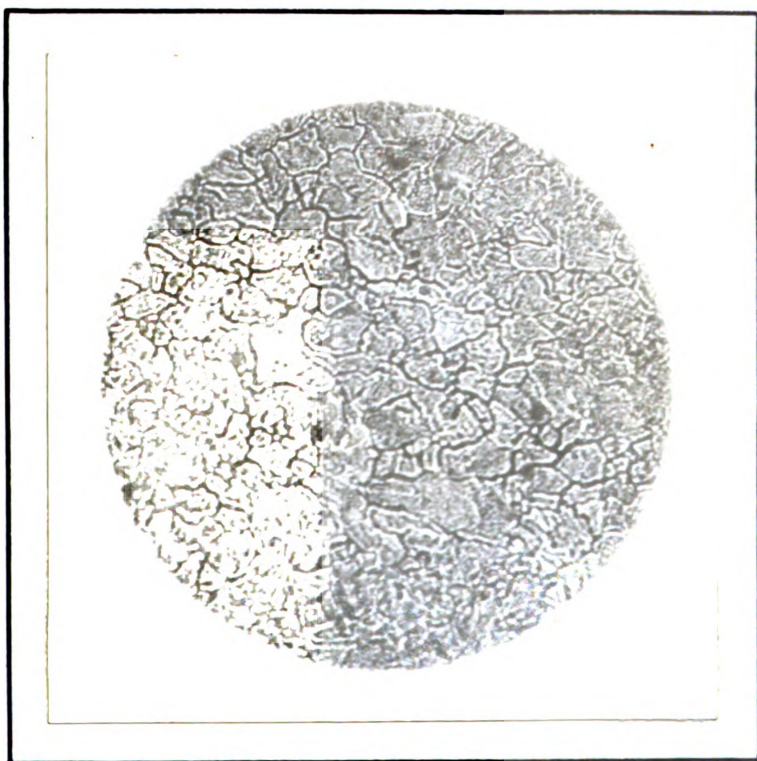
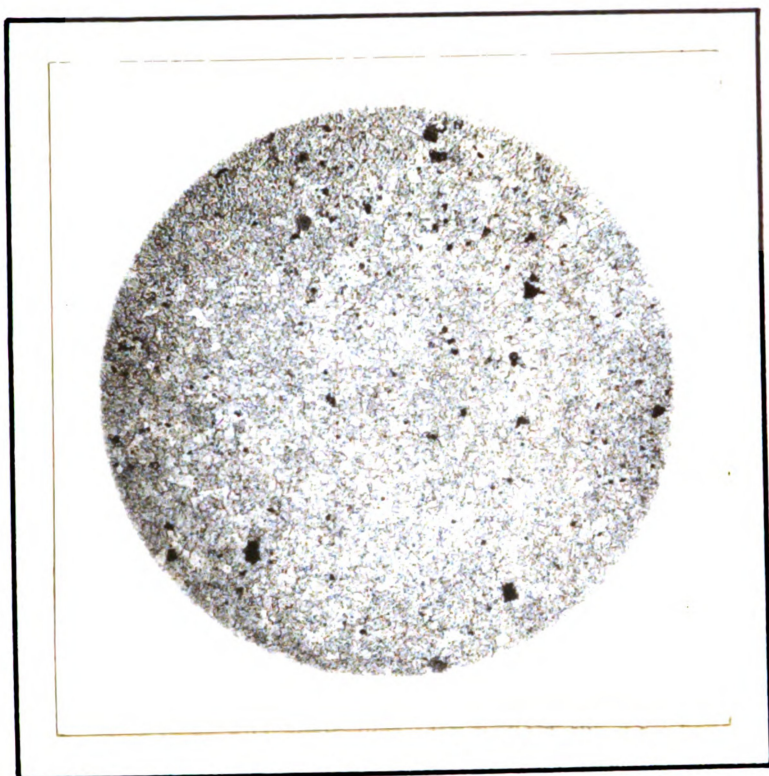
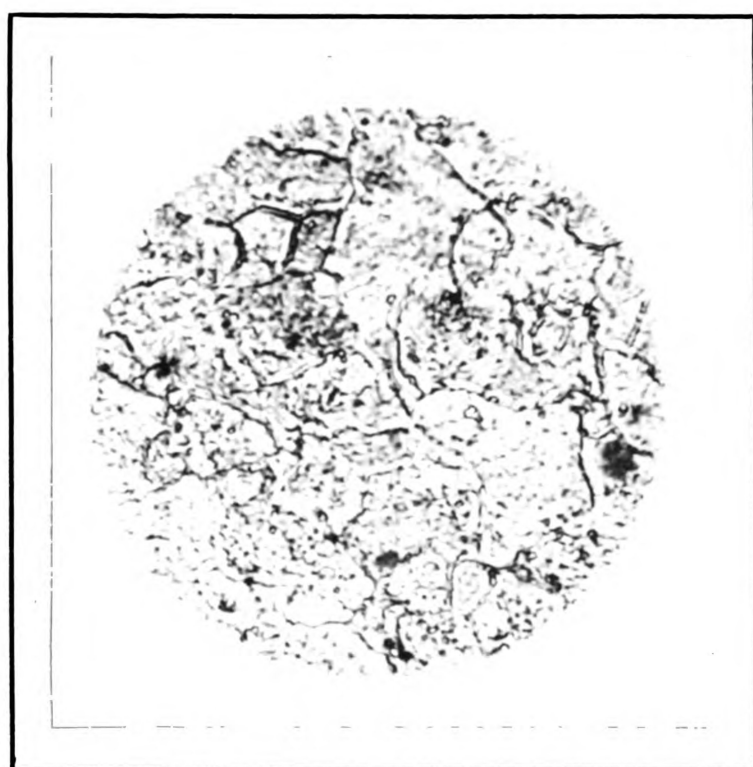


Figure 3
Electron micrograph 1000 X
Same as Fig. 7



was heated up into the critical range and the other was formed as the specimen cooled. Figures 10 and 11 are photomicrographs of the same surface as above at 100 X and 500 X respectively. The entire surface appears to be quite rough. The darker portion, or where there appears to be more boundaries and where there seems to be more concentration of the globule formation, was the area originally occupied by the pearlite.

Film Number 4, which was taken as the specimen was heated from 850 F to 1560 F, shows the change in structure as it took place on the surface. There is no evidence of a wave passing over the polished surface as it passed through the A_3 transformation. The change is gradual. The heat etch appears very faint at first and as the temperature increases it appears more distinct and shows better detail.

Film Number 5 was taken as a sample of medium carbon steel was heated from 450 F to 1550 F. Before this specimen was placed in the vacuum furnace it was etched with a 2% solution of nital. Figure 13 is a photomicrograph of the specimen as it appeared before being placed in the furnace. The first part of the film shows the structure as brought out by the nital etch. It shows the pearlitic areas and the grain boundaries the same as in Figure 13. As the specimen was heated the pearlitic areas disappeared and the boundaries brought out by the etchant became indistinguishable from those caused by heating. The structure of the specimen when at 1500 F did not show any relation to the original structure.

Figures 14, 15, 16 and 17 are photomicrographs taken at 100 X,

Figure 10

Photomicrograph 100 X

Heat etched, cooled from

1560° F

Figure 11

Photomicrograph 500 X

Same as above

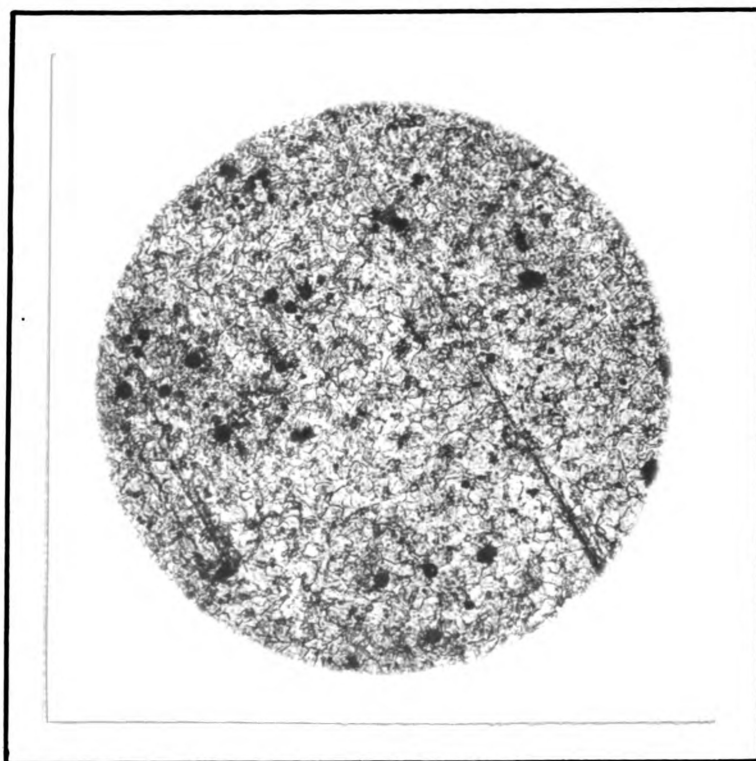
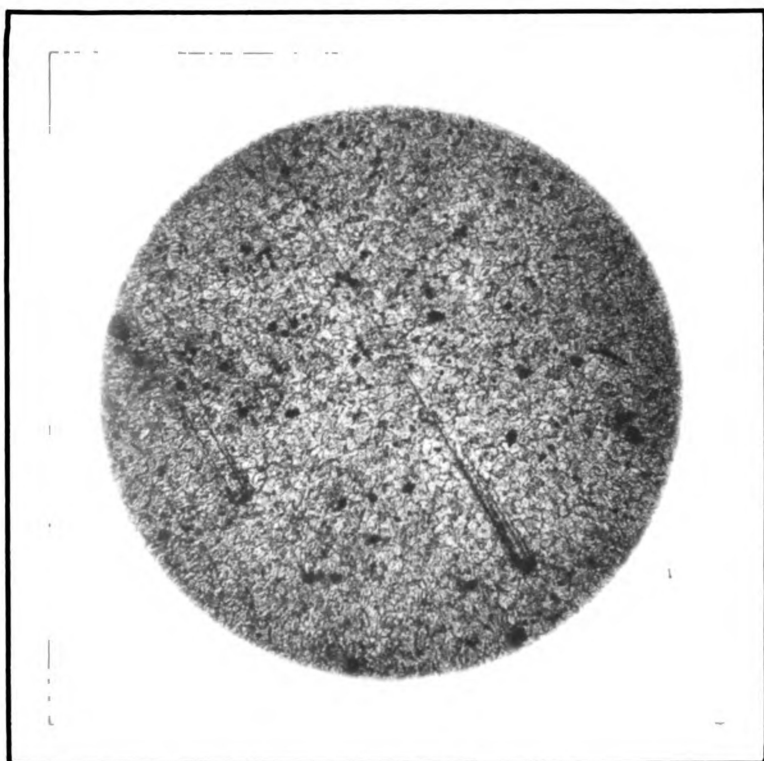


Figure 12

Photomicrograph 1000 X

Same as Fig. 13

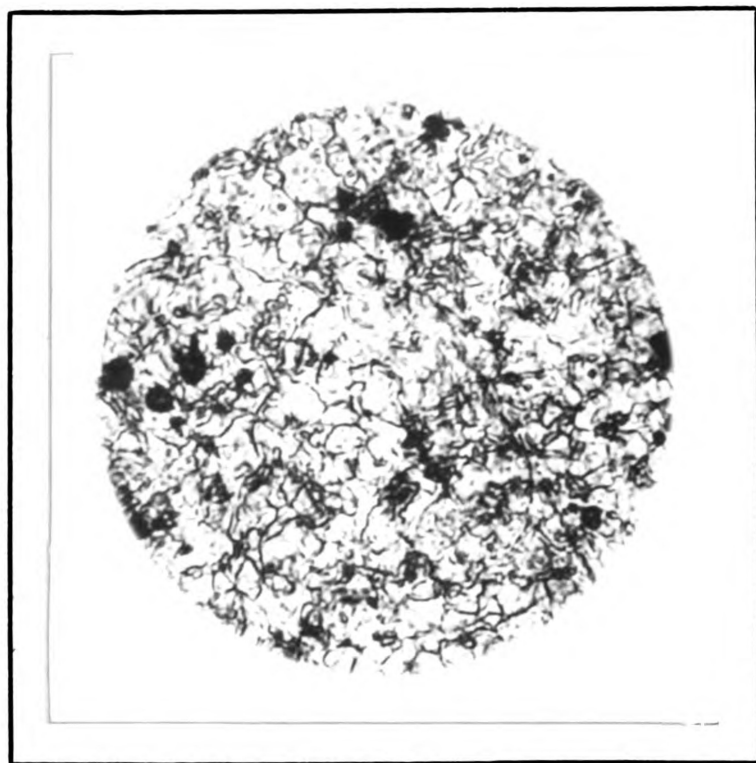


Figure 13
Photomicrograph 100 X
Etched 1% Nitric

Figure 14
Photomicrograph 100 X
Same as fig. 13 after
heated to 1510° F in a
vacuum.

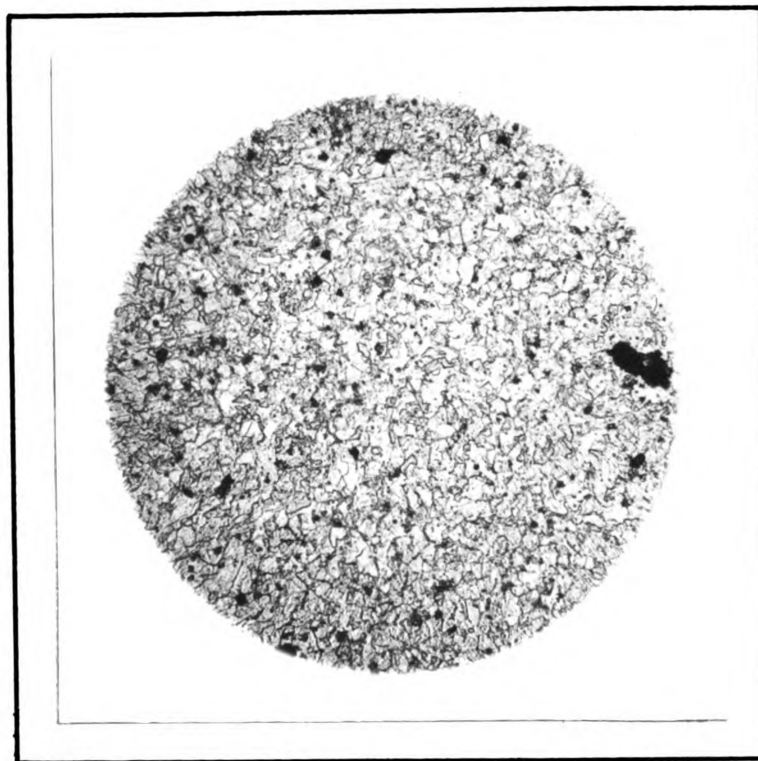
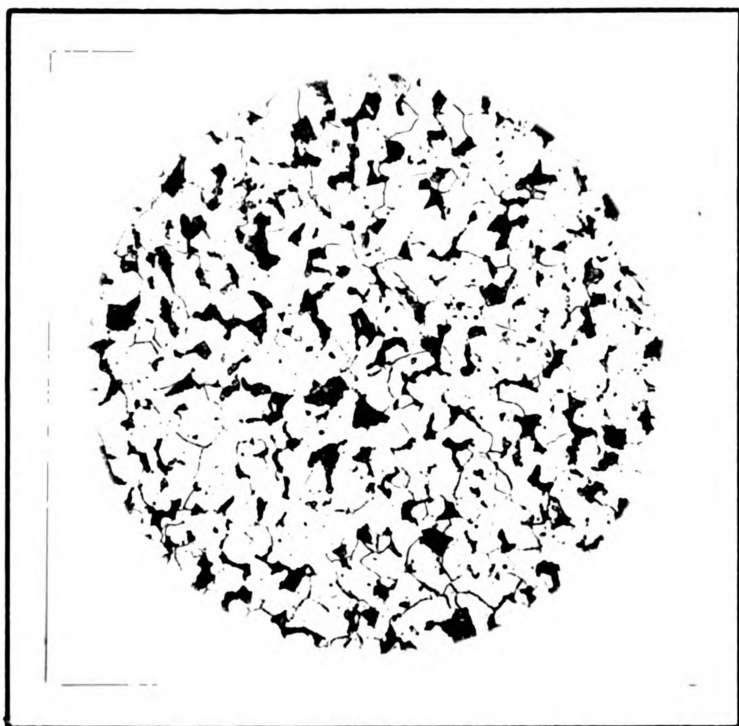
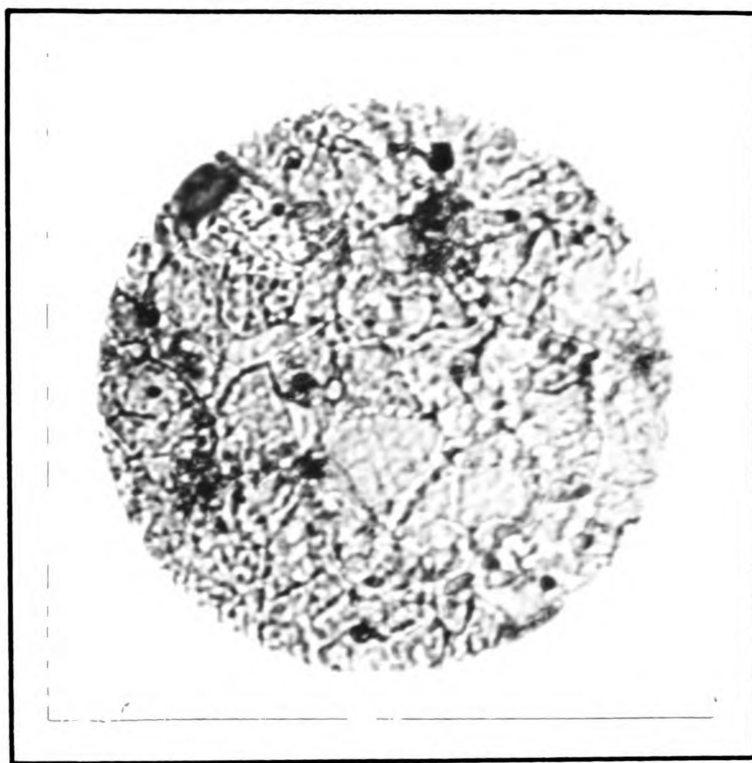
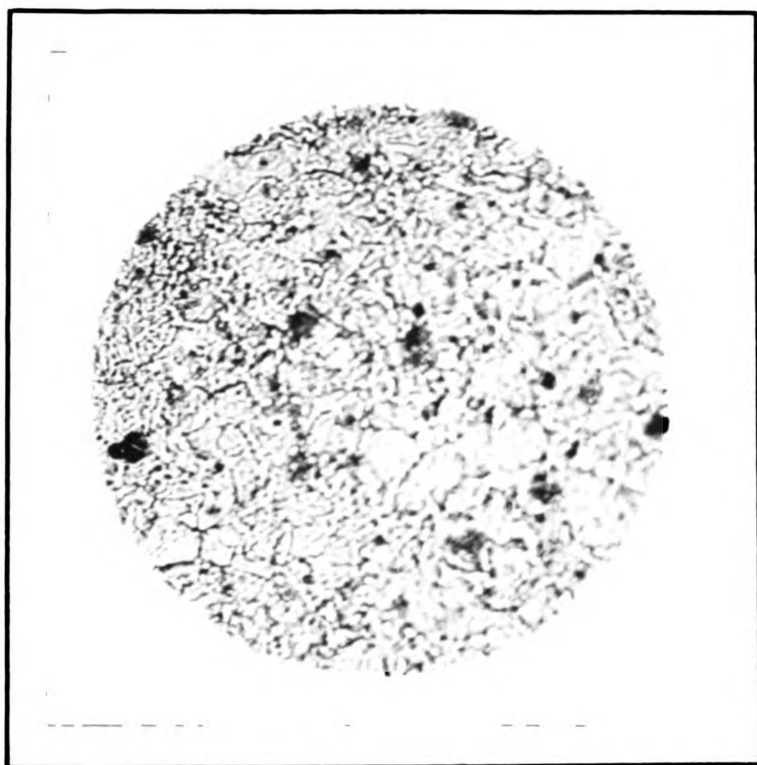
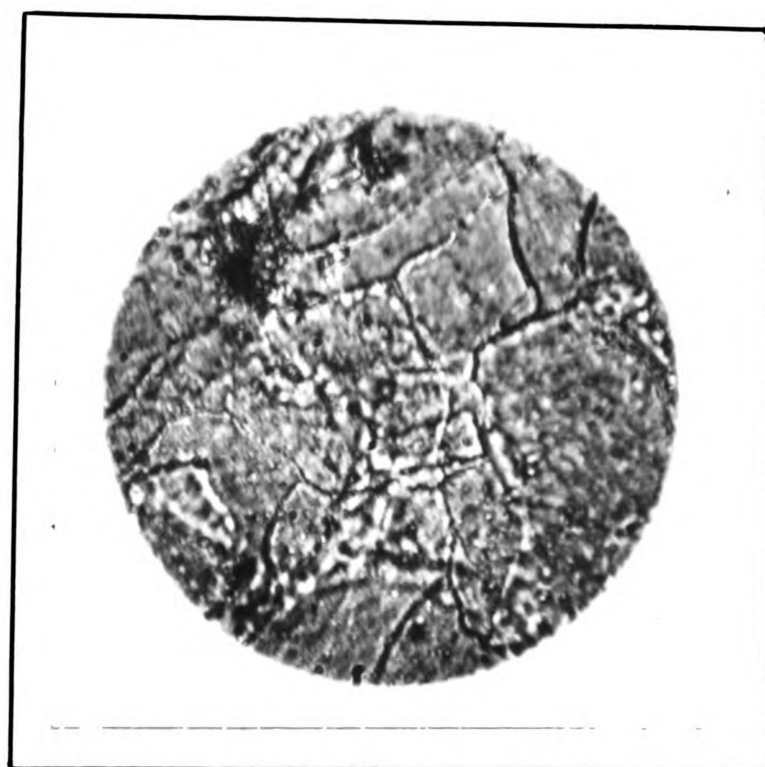


Figure 15
Photomicrograph 560 X
Same as in Fig. 14

Figure 16
Photomicrograph 1360 X
Same as above





500 X, 1000 X, and 2000 X respectively, after the specimen had cooled to room temperature. The structure shown in Figure 14 bears no relation to that as shown in Figure 13, which gives evidence that a reaction had taken place at the surface. At the elevated temperature the polished surface is silver-white and there is no pearlitic formation. This may be due to the fact that all the pearlite has changed to austenite. After the specimen was cooled, the surface had the same silver-white appearance but no pearlite areas. This indicated that the surface of the specimen has been decarburized.

Further evidence of this decarburization is shown in Figure 18, a photomicrograph taken at 150 X of a medium carbon steel after being heated in a vacuum at 1560 F and cooled to room temperature. This photomicrograph also shows the globular formation in the grain boundaries and in the interior of the grains. Figures 19 and 20 are photomicrographs of the same area taken at 500 X and 1000 X.

Film Number 6 is a record of the change in the structure of the polished surface of a steel sample. It is a continuous picture taken as the specimen was heated from 900 F to 1560 F. At the start of the film the surface of the specimen was entirely devoid of detail except for the polishing scratches, but as the temperature increased a faint net-work formed the same as in the preceding trials.

Film Number 7 was taken in the same manner as the others.

Figure 13

Photomicrograph 100 X

Rest etched. Cooled from

1570° F

Figure 13

Photomicrograph 500 X

Same as Fig. 12

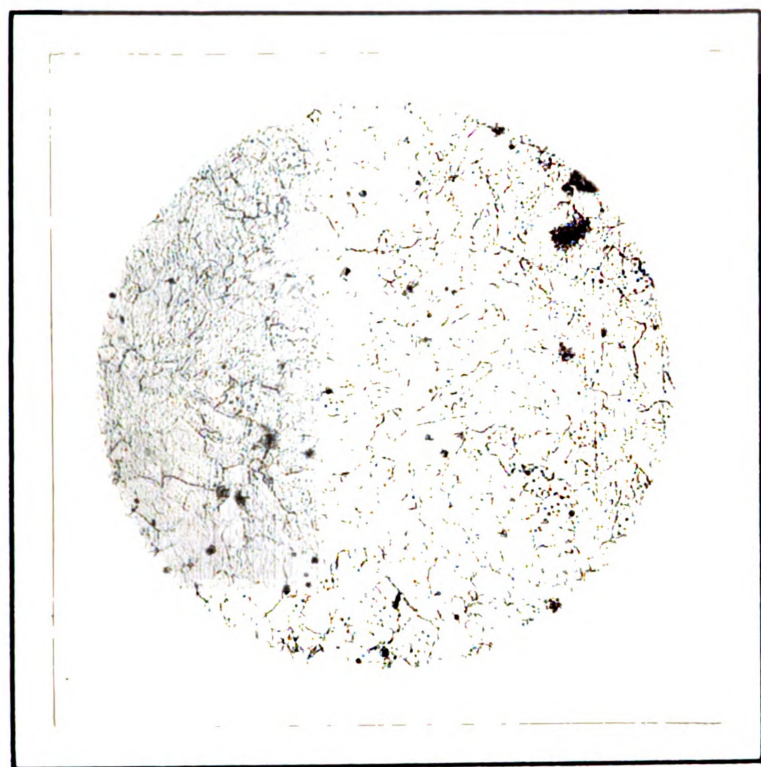


Figure 10

Photo 1000, July 1910

Same as Fig. 13



It shows the surface changes as the specimen was heated from 800°F to 1500°F. The film shows fairly good detail except for the end portion, which is blurred due to vibration. In most all of the films obtained, some frames were lighter than others due to unavoidable changes in the intensity of the light source which was operating on A. C. current. This seemed to be more prevalent when partly consumed carbon electrodes were used in the arc lamp.

Figure 20 is a photomicrograph taken at 100 X of the specimen after it was removed from the vacuum furnace. The surface was silver-white and showed rather fine grain structure. It appeared similar to ferrite, there being no pearlitic areas in evidence. The grain boundaries are much wider than those which are brought out by some acid etchant. Figures 22 and 23, taken at 500 X and 1000 X respectively, show the grain boundaries in much better detail. Figure 23 shows very clearly the double net-work and also the globular formation in the grain boundaries. It shows that the spheroids are much more concentrated in the grain boundaries, if they are such, than through the interior of the grains. In the second set of grain boundaries there is no evidence of this globular formation. The boundaries appear similar to those brought by a liquid etchant. The two different sets of grain boundaries are apparently independent of each other. If the metal is crystalline or granular in nature and the boundaries of these grains are visible through the microscope, there must have been two reactions or changes which took

Figure 81

Photomicrograph 100 X

Heat etched 0.4 O-etch

Figure 82

Photomicrograph 500 X

Same as above.

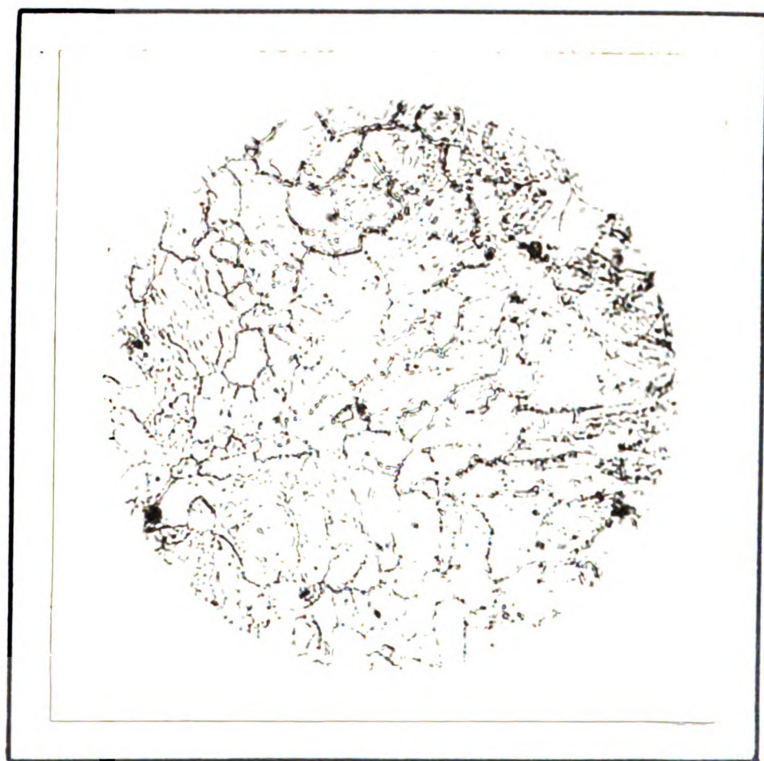
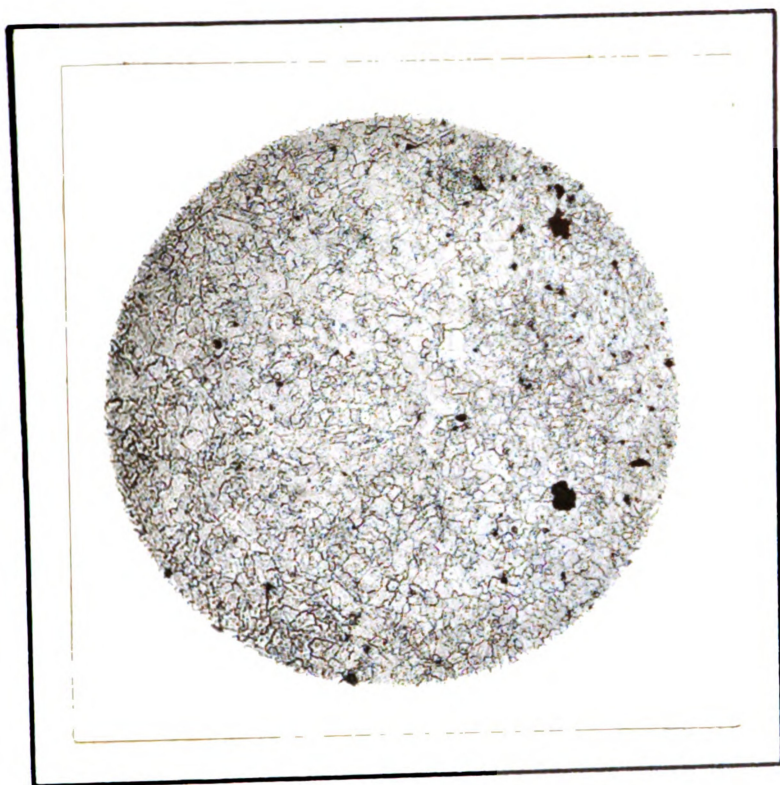
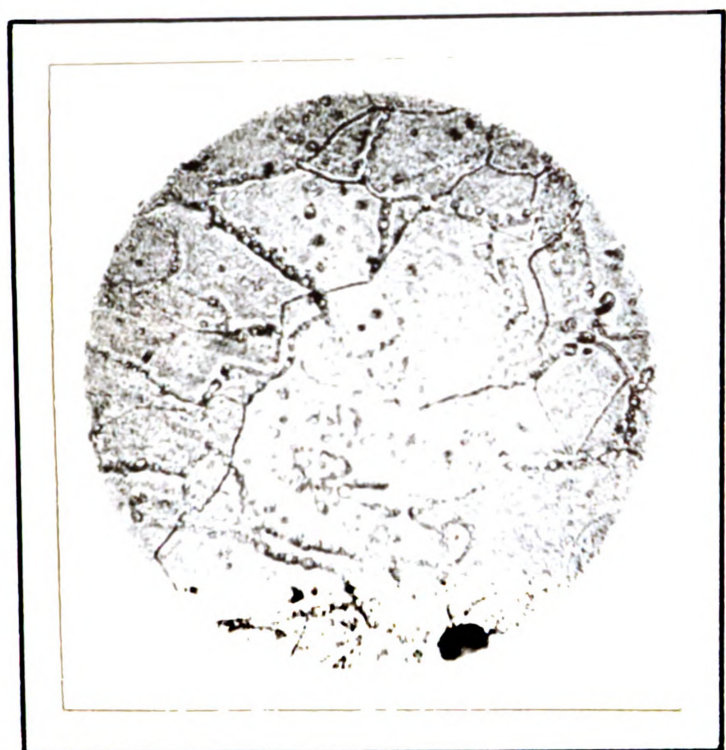




Figure 53
Photomicrograph 100 X
Gravel #1

Figure 54
Photomicrograph 4500 X
Gravel #2 above



place in order to give the two net-work formations.

Figure 24 and 24a, photomicrographs taken at 4500 X of the same specimen as in Figures 21, 22 and 23, show more clearly the double net-work formation, the independency of the two, and the widened condition of one. Figure 25 is a photomicrograph of the same sample after it had been etched in 3% solution of picric acid for several seconds. The wide set of grain boundaries did not seem to be effected by the etchant but the other set seemed to be attacked and brought out more clearly and sharply. This indicated that the carbon had been removed from the wide grain boundaries.

The structure of a specimen etched with a 2% solution of nital is shown in Figure 26. After taking this photomicrograph, the specimen was repolished and heated in a vacuum until a net-work could just be seen. Then the specimen was cooled in the vacuum to room temperature and examined. Figure 27 shows the net-work formed. The net-work formed by heating, or the heat-etch, does not appear to have any relation to the structure as brought out by the etchant. The temperature of the specimen at which the net-work appeared was 1100° F. The sample was lightly polished with levigated alumina to remove the heat etch, and then etched with a 2% solution of nital. Figure 28 shows the structure which is very similar to that shown in Figure 27, but it has no relation to that shown in Figure 26. As there is no relation between the structures shown in Figure 26 and 28, but both were brought out by etching with nital, there must have

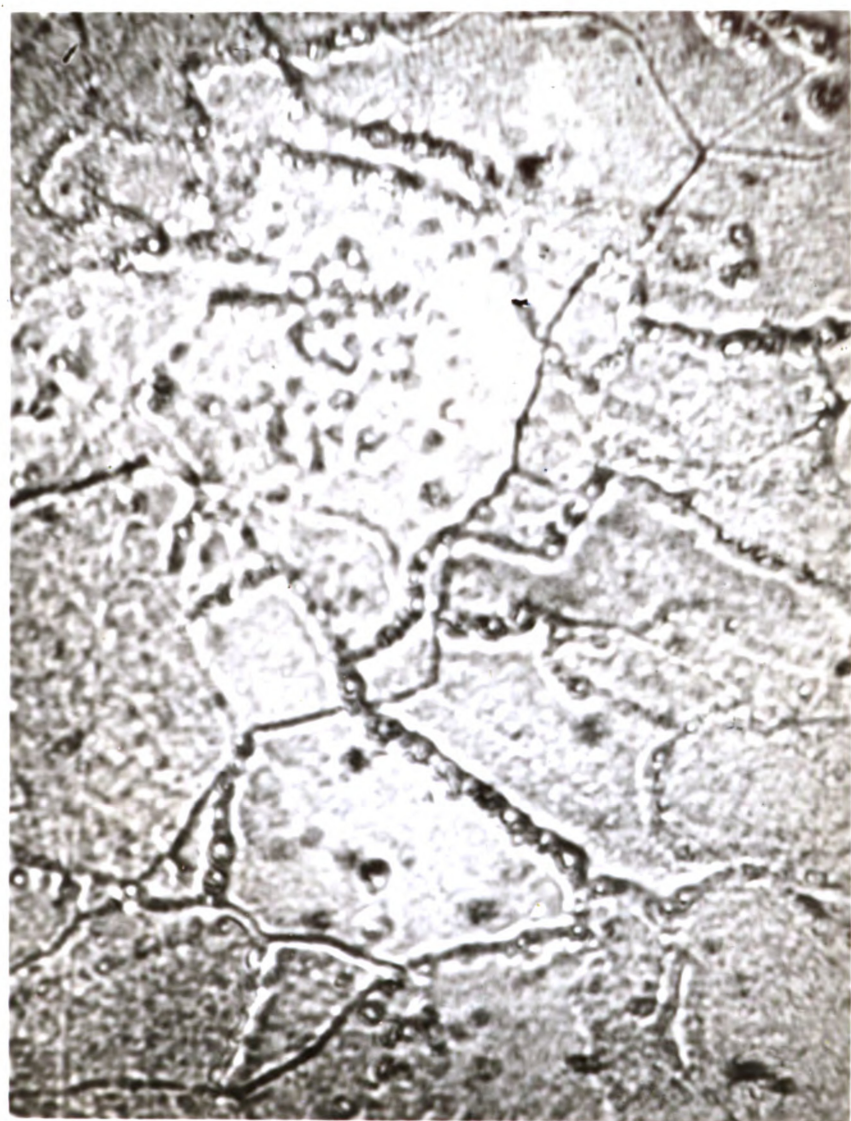


Figure 55

Photomicrograph 4500 X

Same as Fig. 51 etched

with Picric Acid.

Figure 56

Photomicrograph 200 X

Etched 2 % Nitric

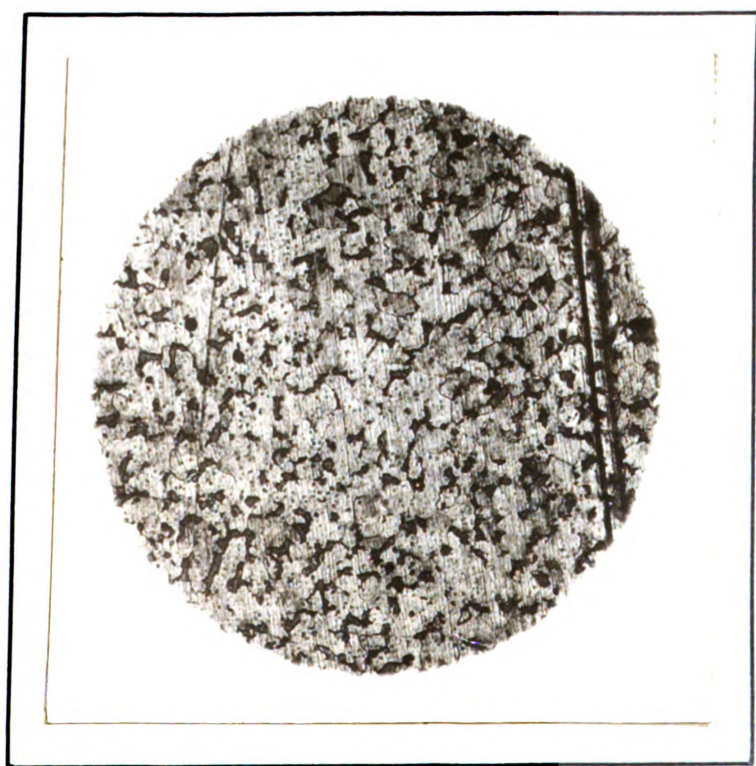


Figure 17

Photomicrograph 200X

Same as in Fig. 56

slight polish. Part

etched in 1% F

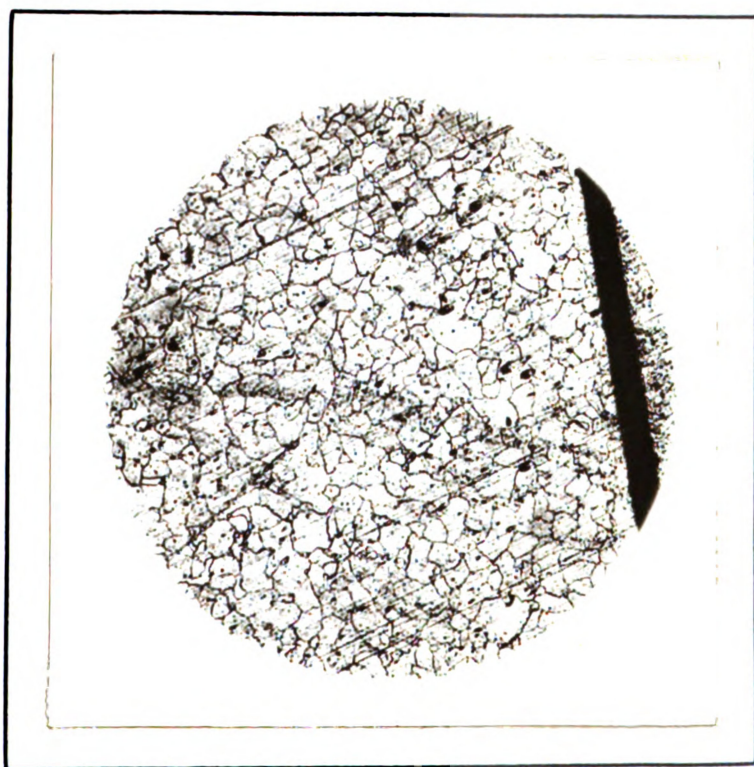
Figure 18

Photomicrograph 200X

Same as in Fig. 57

slight polish and

etched in 1% nitric.



been some reaction which took place when the specimen was heated in the vacuum. This reaction must have taken place below the critical range because the highest temperature of the specimen was 1100° F, unless the temperature at which the A_3 transformation takes place is lowered to 1100° F when a sample of steel is heated in a vacuum. Figure 29 is a photomicrograph of the same specimen as in Figure 23 after it had been more deeply polished and etched with nital. The structure is not the same as in the preceding micrograph, but there are some small pearlitic areas similar to those in Figure 26. As there has been a change in the surface metal and there is no evidence of carbon after the specimen was heated in a vacuum, the carbon must have been volatilized off. In Figure 29 there is no evidence of a second net-work formation or grain structure.

Film Number 8 was made as a specimen of medium carbon steel was heated in a vacuum from 900° F to 1450° F. It shows the net-work formation as it started to form at about 1000° F and continued until the specimen reached 1450° F when there seemed to be no further change. It was attempted to obtain pictures as the specimen cooled, but this portion of the film proved unsatisfactory. The specimen contracted rather rapidly and the microscope had to be continually readjusted.

The magnification on all films is approximately 100 X. This was determined by placing a stage micrometer on the top of the

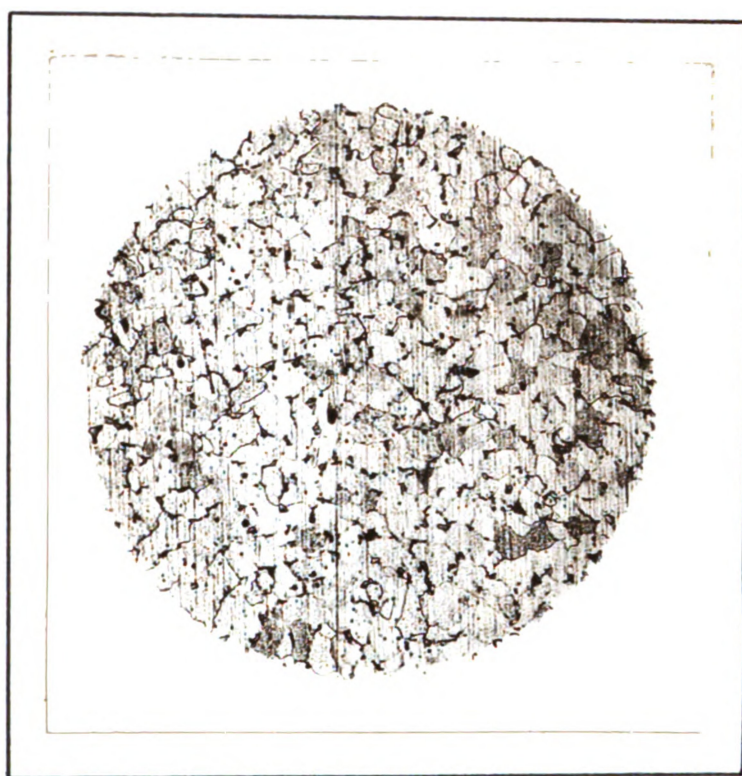
Figure 10

Photomicrograph of 0.7

microns in Fig. 10

slight relief, etched

0.5 microns



furnace and taking a short exposure. The ruled lines on the micro-meter are 0.1 mm. and 0.01 mm. apart and the film shows them to be about 1 cm. and 1 mm. apart respectively, indicating a magnification of approximately 100 diameters.

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DISCUSSION

If iron is cast, it forms grains of gamma iron above 1670°F crystallizing with a face-centered cubic lattice. On cooling, gamma iron changes completely into a body-centered lattice at about 1670°F , and this transformation gives rise to entirely new grains. If the iron is kept below 1670°F , it behaves much like any other metal in a similar temperature range. On heating above about 1670°F , however, the structure again changes completely into new grains of gamma iron. Every time iron is caused to change from one crystal lattice to the other a new set of grains is produced. Thus every time a sample was heated up in a vacuum there was a complete change from the face-centered lattice of the alpha iron to the body-centered lattice of gamma iron if it passed the critical range. (8)

The properties of iron are greatly influenced by small amounts of carbon. As most of the specimens used in this work contained 0.4 percent carbon, the temperature at which this change takes place is lowered to about 1390°F . When this change takes place the metal expands because the body-centered arrangement of the atoms is not so closely packed as the face-centered arrangement. During the volume change the metal cools a slight degree. If a curve is plotted from time temperature data taken while a specimen is being heated at a constant rate, there will be a place in the curve where the temperature will remain constant or rise at a slower rate. This shows that the heat applied is being absorbed by the

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specimen more rapidly than before, and that a change is taking place in the metal. This is shown by the two preceding curves, plotted from data taken while a specimen was heated in a vacuum at a comparatively slow rate. The curves show that the change takes place between 1100° F and 1250° F while theoretically if the metal was heated in air this change should take place from 100° to 200° F higher. This gives evidence that pressure effects the transformation temperature. Many unsuccessful attempts were made to obtain time temperature data as the specimen cooled, but the rate of cooling was too rapid and it could not be controlled with the apparatus used. Parker, in his work, showed that the transformation temperature was lowered when the pressure was reduced.

When a specimen was heated in a vacuum a distinct net-work or set of grain boundaries was formed which had no relation to the structure of the specimen before heating. Thus a change must have taken place. It might have been a volume change, but as the temperature had not reached the critical range so that the space lattice would change, there must have been some other reason for it.

When foreign atoms dissolve in a metal to form solid solutions, if the lattice expands or the cube edge is increased, the solid solution is termed interstitial. If it is of the substitution type, however, it expands if the volume of the foreign atoms is greater than the volume of the atoms of the solvent, and contracts if the volume of the foreign atom is smaller.

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In a substitution solid solution the atoms of the solute may replace some of the atoms of the solvent in the space lattice. In an interstitial solid solution the atoms of the solute may be located at random, except at the lattice points within the space lattice of the solvent. There is a small amount of carbon in solution in ferrite or alpha iron. Carbon in iron forms an interstitial solid solution. As the surface of the sample is decarburized when heated, the space lattice of the iron remaining must have a decreased cube edge and thus a decrease in volume. The interior of the specimen is not decarburized and when heated would increase in volume due to normal expansion. These volume changes acting in different directions might account for the buckled appearance of the surface below the critical range.

It is held that it is impossible for an intermetallic compound, as such, to dissolve in a metal, as there is no room within the space lattice for admission of molecules. The carbide Fe_3C could not, as such, dissolve in iron. The carbon must be present in atomic dispersion which implies the dissociation of Fe_3C previous to the formation of a solid solution and necessarily the formation of that compound as carbon comes out of solution. As carbon is almost insoluble in alpha iron or ferrite, it must be in the carbide form, and in normalized steel it is located in the grain boundaries and pearlite areas. When a sample of steel is heated in a vacuum decarburization takes place. This may

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account for the globular formation in the grain boundaries as shown in Figure 24. If the carbon leaves the metal as carbon, and not as the carbide, it must leave the iron that it was combined with in the grain boundaries. If this is the case it is apparent that the globular formation would be concentrated in the grain boundaries because that is where the carbide is concentrated.

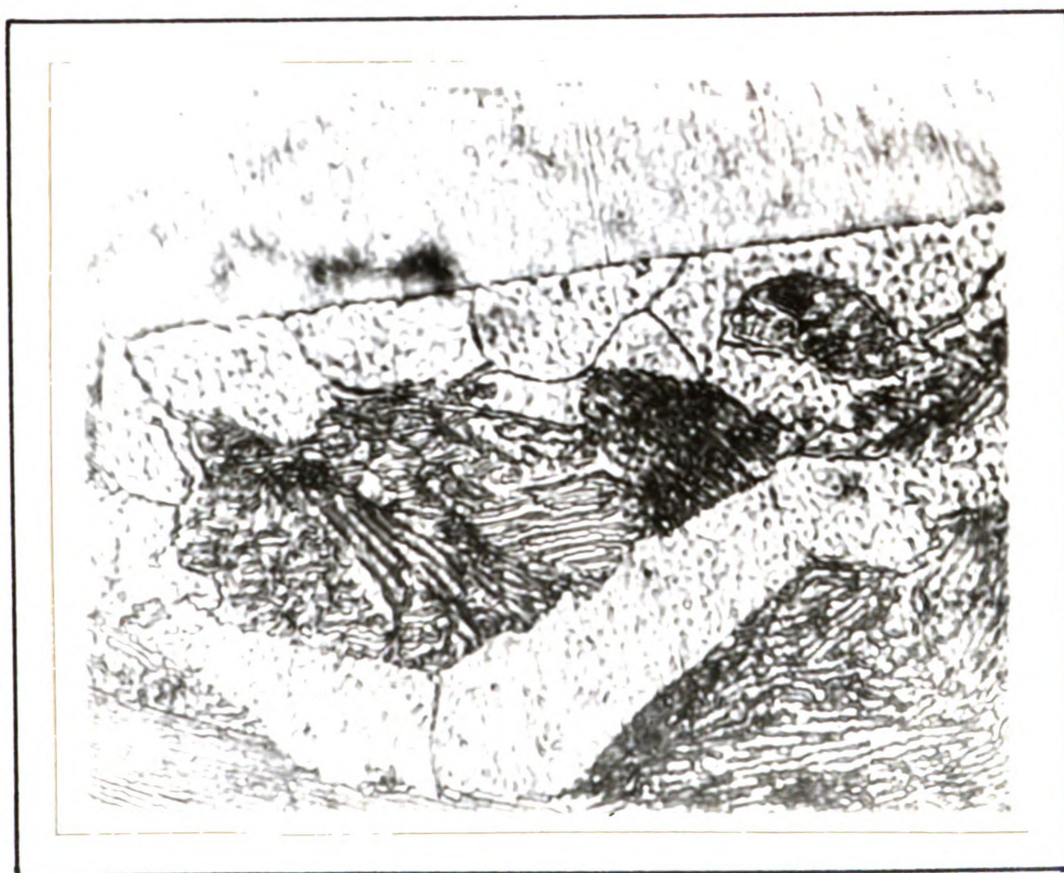
When steel is heated the carbide must become more and more unstable until the transformation range is reached and it decomposes. The carbon is in atomic dispersion so that it can go into solution in gamma iron. As the carbon is in atomic dispersion and unstable at the transformation range, decarburization would take place more rapidly than if the temperature were above the transformation range, since the carbon is then in solution in the gamma iron and more stable. The assumption is made that there is a certain amount of carbon that goes into solution.

After a sample of steel was heated in a vacuum above the A_3 transformation and cooled there were two grain structures present. One had wide grain boundaries and the other small and sharp boundaries. When observing a specimen as it was heated, there was but one visible structure, the grain boundaries of which became wider as the temperature increased. The widening of the boundaries may have been due to the decarburization. If the second net-work was formed as the specimen was heated, it should appear wide as well as the first, but as it did not, it may have formed due to the recrystallization above the transformation point. The second net-work may

also have formed as the specimen cooled. It would form a different pattern because the composition of the surface was not the same when the specimen cooled as it was on heating, due to the loss of carbon. Figure 30 is a photomicrograph taken at 2500 X of a section perpendicular to a heat-etched surface. Nickel was plated on the surface to preserve the existing structure. The decarburized layer and roughened surface are plainly visible. The roughened condition of the surface may be due to the following: volume change, decarburization, volatilization of the surface metal, or to the escaping of "occluded" gases.

Figure 20

Section 1, on the left to a flat
etched surface, 2100 Å. Nickel
is still visible on the sur-
face. The grain boundaries
are still clearly visible.
Picture by
D. H. R.



SUMMARY

This method of investigation can not be readily applied to alloys because of the surface changes. The surface may not show the condition of the interior of the metal. It could be applied more easily to pure metals and much better results obtained.

When a sample of steel passed through the A_3 transformation there was no evidence of a wave passing over the surface.

There are two distinct and independent net-works or grain structures formed. One as the sample was heated and the other as it cooled.

A thin layer of the surface metal is decarburized. Decarburization takes place more rapidly within the critical range.

Two suggestions that might help in carrying out further investigation are: (a) A variable resistance should be used so that the heating and cooling rate can be more closely controlled, and (b) a means of preventing the camera from vibrating due to the motor and gear train, should be devised. This might be accomplished by placing small pieces of rubber sponge between the camera and the camera support.

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10. Wiester, H. J. Martensite Forms Instantly, Metal Progress Vol. XXIII No. 2, 1933.

FILM INDEX

- No. 1 Shutter speed No. 4; 0.3 smoked glass, temperature range 950° F to 1400° F. Unetched sample of 0.4 carbon steel. Arc lamp. Green filter.
- No. 2 Shutter speed No. 4; 0.3 smoked glass, temperature range 800° F to 1500° F. Sample of 0.4 carbon steel unetched. Arc lamp. Green filter.
- No. 3 Shutter speed No. 4; 0.3 smoked glass, temperature range 850° F to 1500° F. Sample of 0.4 carbon steel unetched. Arc lamp. Green filter.
- No. 4 Shutter speed No. 4; 0.3 smoked glass, temperature range 850° F to 1550° F. Sample of 0.4 carbon steel etched 2% nital. Arc lamp. Green filter.
- No. 5 Shutter speed No. 4; 0.3 smoked glass, temperature range 800° F to 1550° F. Sample of 0.4 carbon steel etched 2% nital. Arc lamp. Green filter.
- No. 6 Shutter speed No. 4; 0.3 smoked glass, temperature range 900° F to 1560° F. Sample of 0.4 carbon steel unetched. Arc lamp. Green filter.

- No. 7 Shutter speed No. 4; 0.3 smoked glass, temperature range 800° F to 1500° F. Sample of 0.4 carbon steel unetched. Arc lamp. Green filter.
- No. 8 Shutter speed No. 4; 0.3 smoked glass, temperature range 900° F to 1450° F. Sample of 0.4 carbon steel unetched. Arc lamp. Green filter.
- No. 9 Shutter speed No. 4; 0.3 smoked glass, temperature range 650° F to 1400° F. Sample of 0.4 carbon steel unetched. Mazda ribbon filament lamp. No filter.
- No. 10 Short film taken at the different shutter speeds using each smoked glass at each speed. Arc lamp as source of light and green filter used.

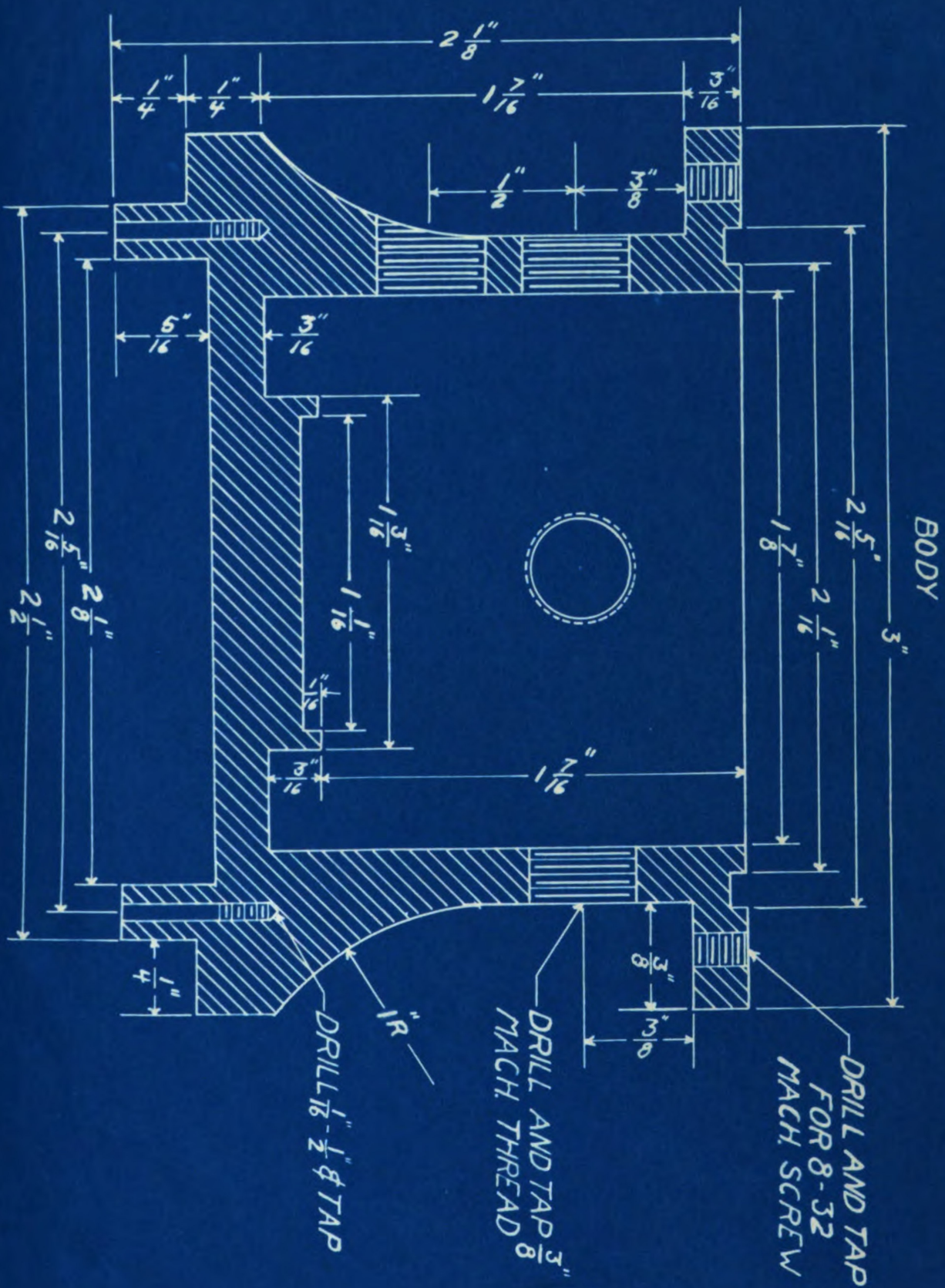
The Number four speed used in all the films takes one frame or one exposure every two seconds. The 0.3 smoked glass allows 70 percent of the light to pass.

All of the above listed films are available at the main office of the Chemical Engineering Department, Room 413 Olds Hall, Michigan State College, East Lansing.

Blue Print

No.1

Section of body



Blue Print

No. 2

Top view of body

1. The first part of the document

2. The second part of the document

3. The third part of the document

BODY



Blue Print

No.5

Top

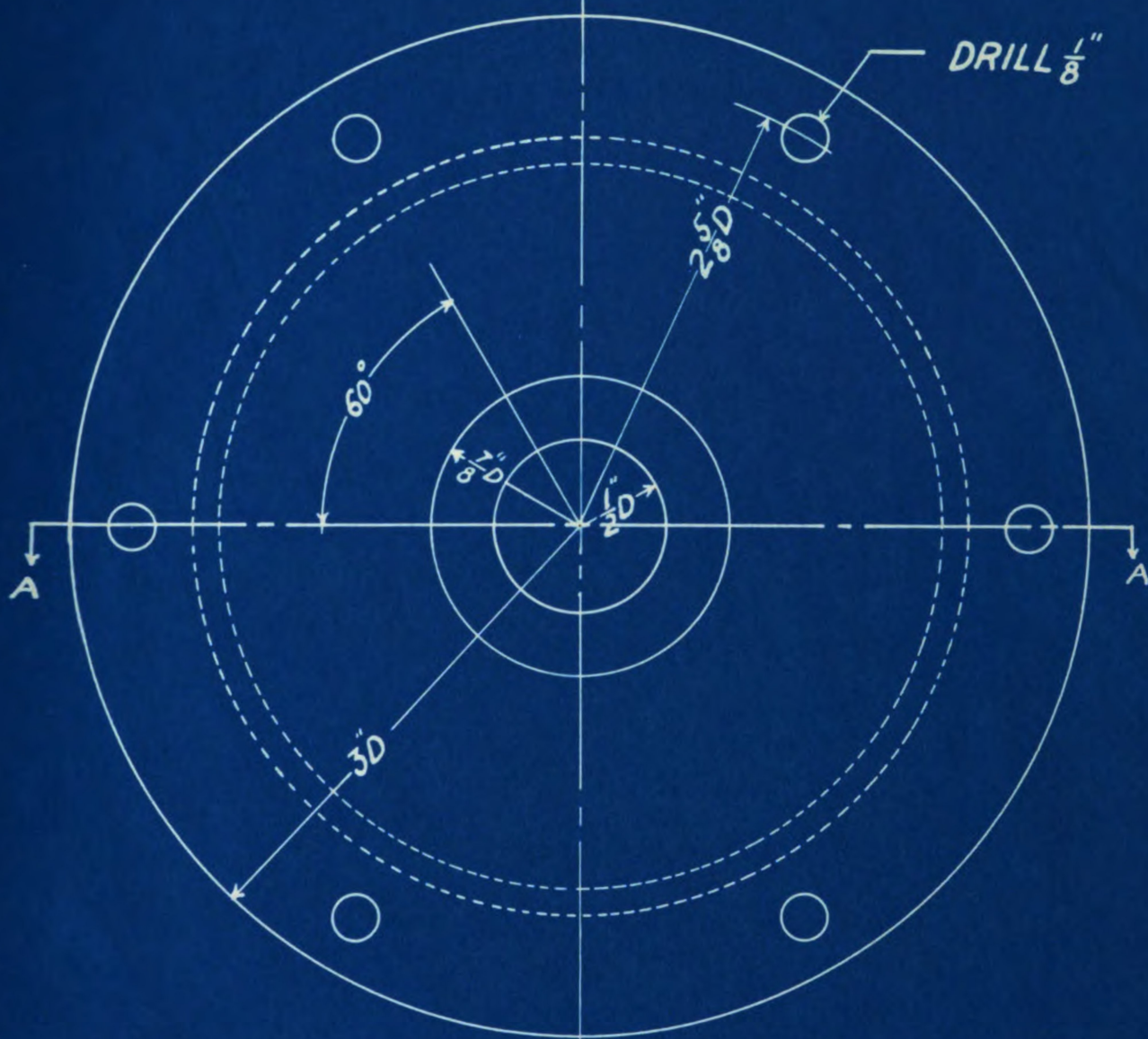
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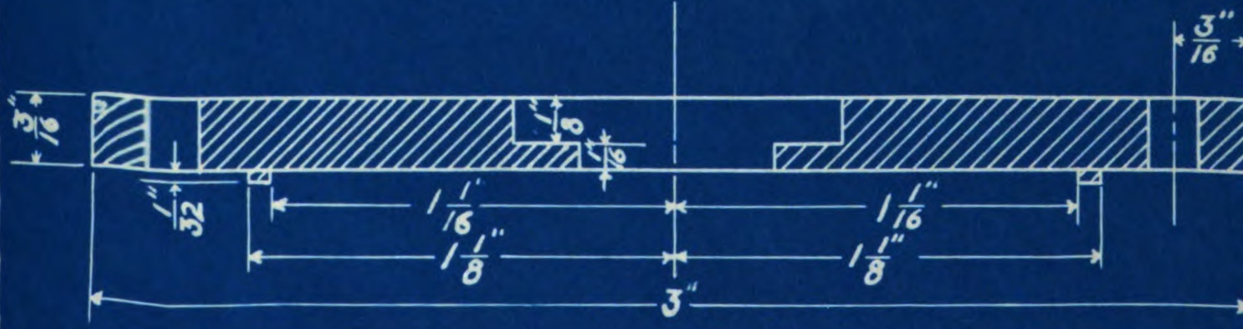
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SECTION-AA

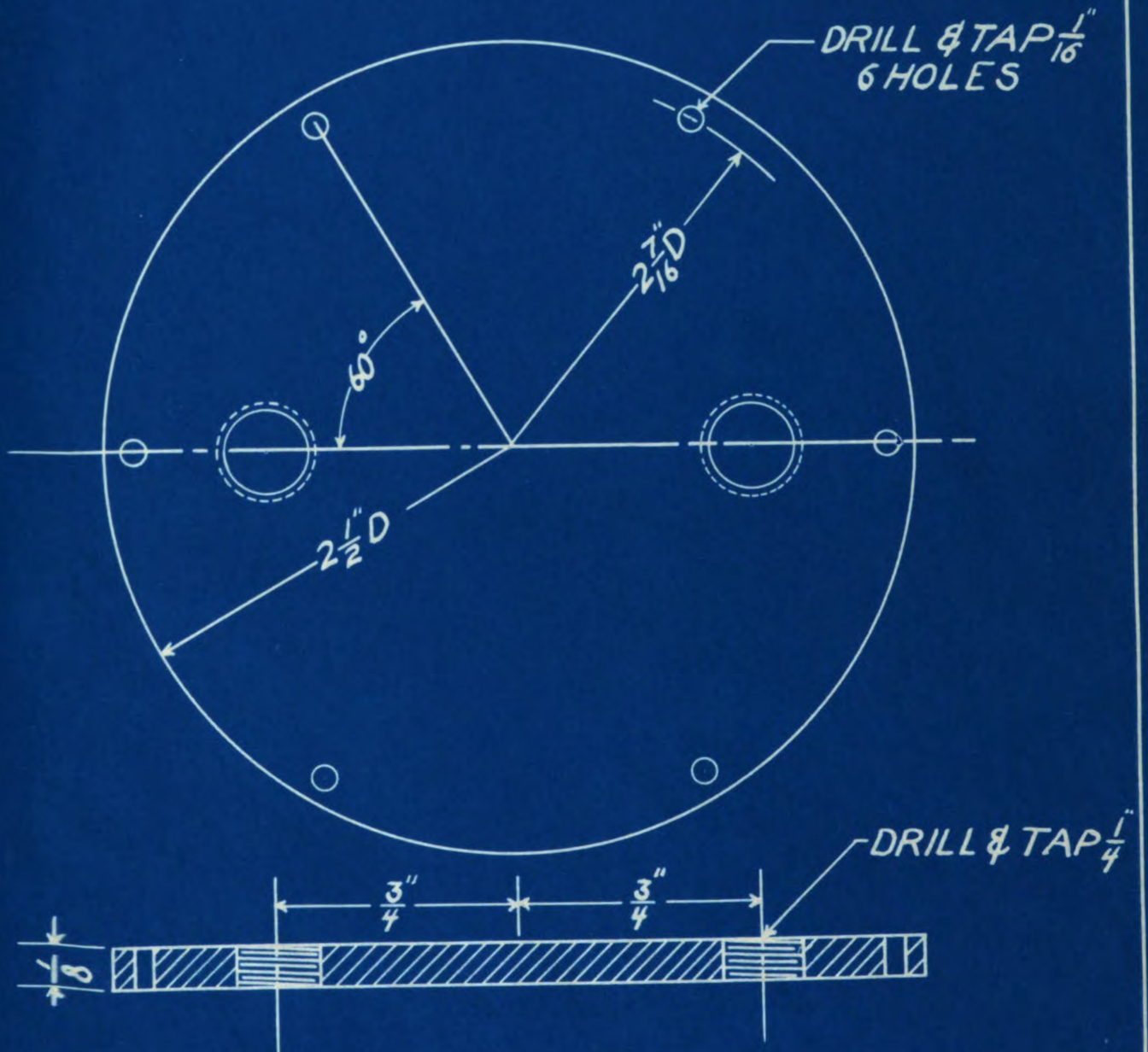


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Bottom

BOTTOM



F.W. Kerr

Blue Print

No.5

Lead*in Plugs

Vacuum pump connection

1000
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Blue Print

No .6

Heating element

Specimen

Specimen Holder

12. 12. 1971

13. 12. 1971

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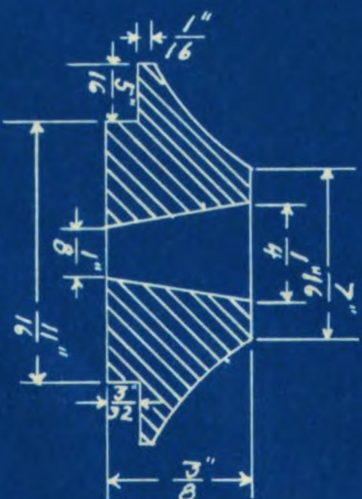
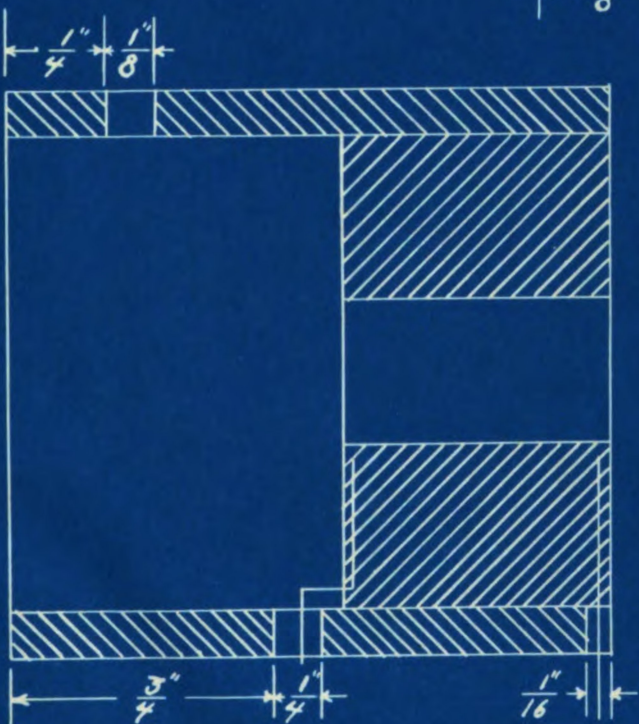
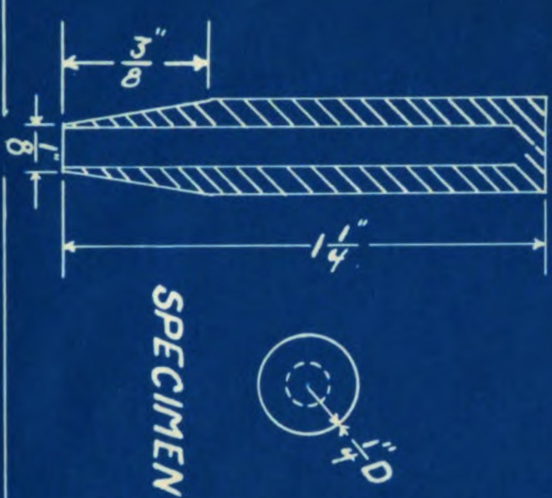
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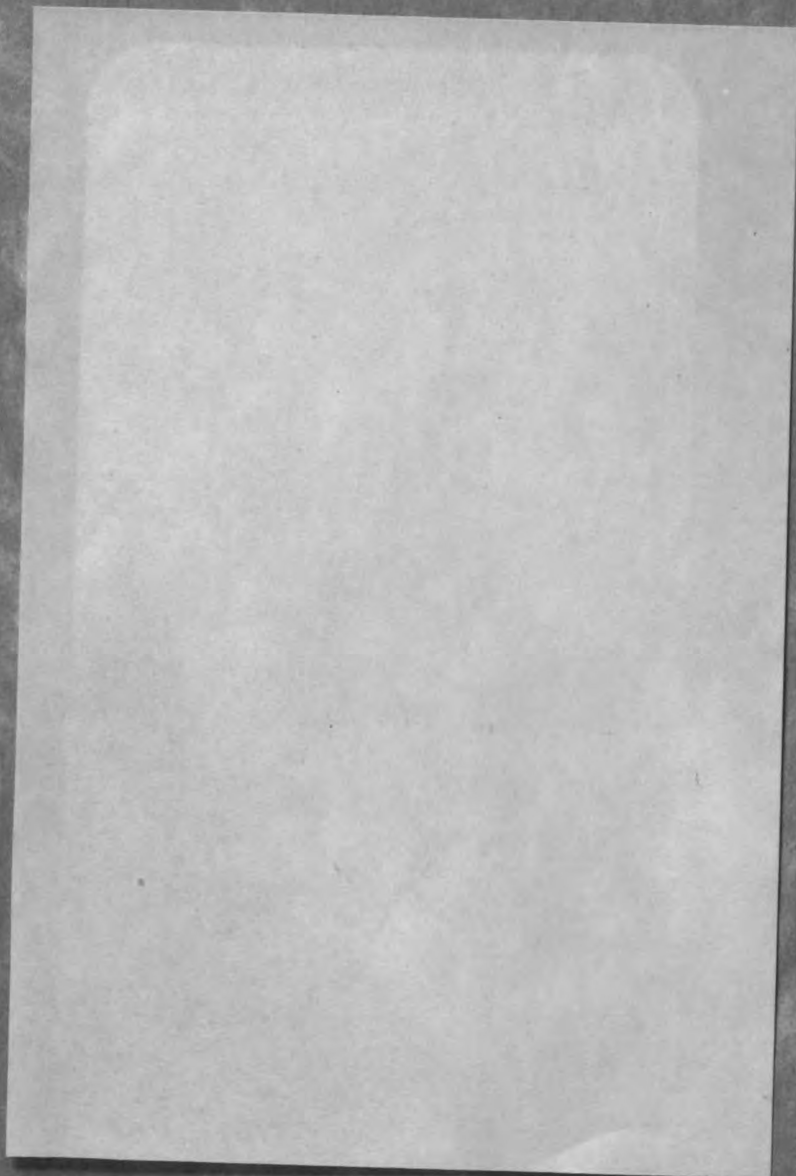
HEATING ELEMENT



SPECIMENHOLDER



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