

File.
FILE COPY

O T T A W A

May 3rd, 1943.

R E P O R T

of the

ORE DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 1398.

An Investigation of the Effect of Overheating on
the Impact Strength of "S-80" Stainless Steel.

IR 1398

209

1764

Abstract.

"S-30" stainless steel was air cooled and slowly cooled from 2250° F. and then re-heat treated by heating to 1800° to 1850° F. for 2, 5, and 10 hours, followed by an oil quench and a draw at 1200° F.

It was found that the impact strength of the steel was seriously impaired by the high-temperature treatment and that even 10 hours at 1800° F. only very slightly restored this value.

The loss in impact strength is thought to be related to the formation of carbides at the grain boundaries at the ferrite-martensite interface. It was found that at normal heat-treating temperatures these carbides were very persistent, ten hours at the heat-treating temperature not being sufficient to completely re-dissolve them.

No conclusive evidence could be found relating to grain size or grain refinement.

A phase diagram and some twenty photomicrographs are presented to illustrate the arguments advanced.

O T T A W A

May 3rd, 1943.

R E P O R T
of the
ORE DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 1398.

An Investigation of the Effect of Overheating on
the Impact Strength of "S-80" Stainless Steel.

Origin of Request and Object of Investigation:

On March 20th, 1943, Wing Commander A. J. Smith, on behalf of the Director of Aeronautical Inspection, Department of National Defence for Air, Ottawa, Ontario, inquired with regard to the effect that high-temperature forging might have on the impact properties of "S-80" stainless steel. A letter expressing a tentative opinion was written from these Laboratories on April 7th, 1943.

In order that this situation might be clarified, a series of experiments was conducted on some "S-80" steel that was available. The present report presents the results obtained in these tests and discusses their implications.

Chemical Analysis of S-80 Steel Used for Investigation:

Table I.

	Obtained	Required
	Per cent	
Carbon	0.13	Not more than 0.25
Silicon	0.45	" " " 0.50
Manganese	0.74	" " " 1.0
Nickel	1.56	Not less than 1.0
Chromium	16.76	16.0 to 20.0
Sulphur	0.02	"
Phosphorus	0.012	"

Program of Investigation:

The following program was drawn up for this investigation (the steel was received in the form of hot-rolled bar stock, fully heat-treated):

Treatment A - Heat to 2250° F.,
Hold for 5 hours,
Air cool,
Obtain a specimen for hardness and microstructure, and
Cut remaining steel into three portions, A₁, A₂, and A₃.

Treatment B - Heat to 2250° F.,
Hold for 5 hours,
Transfer to furnace at 1300° F.,
Hold for 5 hours,
Air cool,
Obtain a specimen for microstructure and hardness, and
Cut remaining steel into three portions, B₁, B₂, and B₃.

Treatments A₁ and B₁ - After Treatments A and B,
Heat to 1600° F.,
Hold for 2 hours,
Oil quench,
Heat to 1200° F.,
Hold for 1 hour,
Oil quench, and
Obtain izod impact values, hardness, and microstructure.

The purpose of this treatment is to determine whether or not a normal heat treatment could remove the effect of a high forging temperature regardless of the rate of cooling from that temperature.

(Continued on next page)

(Program of Investigation, cont'd) -

Treatments A_2 and B_2 - After Treatments A and B,
Heat to 1850° F.
Hold for 3 hours,
Air cool,
Reheat to 1800° F.,
Hold for 2 hours,
Oil quench,
Heat to 1200° F.,
Hold for 1 hour, and
Oil quench.

This heat treatment was followed because it approximated one, given in previous correspondence, about which there was some argument.

Treatment A_3 - After Treatment A,
Heat to 1800° F.,
Hold for 10 hours,
Oil quench,
Heat to 1200° F.,
Hold for 1 hour,
Oil quench, and
Obtain impact properties, micro-structure, and hardness.

This heat treatment was carried out to determine the effect of time at the hardening temperature.

Summary of Results:

The impact and hardness values and the key to the photomicrographs are given in Table II. Due to the limited amount of material available it was only possible to make single-notch bars from A_1 , A_3 and B_1 . A_2 and B_2 were three-notch bars.

Table II.

Test No.	Load : foot pounds	Vickers : hardness, 30-kg. load	X100	X1000	X2000	X1000 carbide etch
As rec.	44.7	282	Fig. 1	Fig. 2	-	Fig. 3
A	-	455	Fig. 4	Fig. 5	-	Fig. 6
B	-	320	Fig. 7	Fig. 8	Fig. 9	Fig. 10
A_1	6	285	-	Fig. 11	-	Fig. 12
B_1	4	279	-	Fig. 13	-	-
A_2	8	280	Fig. 14	Fig. 15	-	-
B_2	7	277	Fig. 16	Fig. 17	-	-
A_3	11	272	Fig. 18	Fig. 19	-	Fig. 20

MICROSCOPIC EXAMINATION:

The following methods of preparation of the specimens for microscopic examination were employed:

Polishing:

For Figures 1, 2, 4, 5, 7, 8, 11, and 13 to 19, the specimens were electrolytically polished.

For Figures 3, 6, 9, 10, 12 and 20, the specimens were polished by hand. This latter procedure was found necessary because the electrolytic polishing preferentially attacked the free carbides at the grain boundaries.

Etching:

To reveal the martensitic and ferritic phases, the following etching procedures were employed:

Specimens for Figures 1, 4, 5, 7, 8, 11, 13, 14, 15, 16, 17, and 18 were etched in the following reagent:

5 c.c. hydrochloric acid.
1 gram picric acid.
100 c.c. ethyl alcohol.

Specimens for Figures 2, 9 and 19 were electrolytically etched as follows:

Electrolyte	-	10 c.c. hydrochloric acid. 90 c.c. ethyl alcohol.
Voltage	-	4 volts.
Current	-	0.4 amps.
Time	-	10 seconds.

The results of these two procedures are identical.

Specimens for Figures 3, 6, 10, 12 and 20 were electrolytically etched as follows:

Electrolyte	-	10 grams tartaric acid. 90 c.c. water.
Voltage	-	6 volts
Current	-	0.15 amps.
Time	-	5 to 10 minutes.

This etching blackens free carbides and also lightly etches tempered martensite but does not attack the martensite phase formed on cooling from above the critical temperature (either in air or by quenching) and not subsequently tempered.

(Continued on next page)

(Microscopic Examination, cont'd) -

A comparison of Figures 1, 4 and 7 reveals that phase redistribution has taken place on heating up to 2250° F. The long continuous stringers of the ferritic phase have been broken up but there is still some evidence of a banded structure. It is not possible, from these photomicrographs, to get any clear idea of the grain size of the steel. However, it does not have the appearance of being excessively coarse-grained and on comparing Figures 4, 7, 14, 16, and 18 there does not appear to be much grain refinement resulting from re-heat treatment after high-temperature treatment. About the most striking effect of the high-temperature treatment has been to partly break up the ferrite bands.

Figures 2, 11, 13, 15, 17, and 19 are presented to show that the structure of the martensitic phase in the metal in its final quenched-and-drawn condition is about the same as in the steel in its original condition. This fact, combined with the hardness values, would tend to indicate that the final tempering temperature chosen was about the same as that used for the original heat treatment.

One of the reasons for the low impact is evident from studying Figures 6, 9, and 10. It will be noted that a carbide network has been formed. This network is more general in occurrence at the martensite-ferrite interface but also exists at some of the grain boundaries in the martensite. When cooled very slowly, as in Test B, this carbide evidently even precipitates within the ferrite. This can be seen from a study of both Figure 9 and Figure 10. The large grains in Figures 6 and 10 are martensite. These did not etch because they were not tempered.

(Continued on next page)

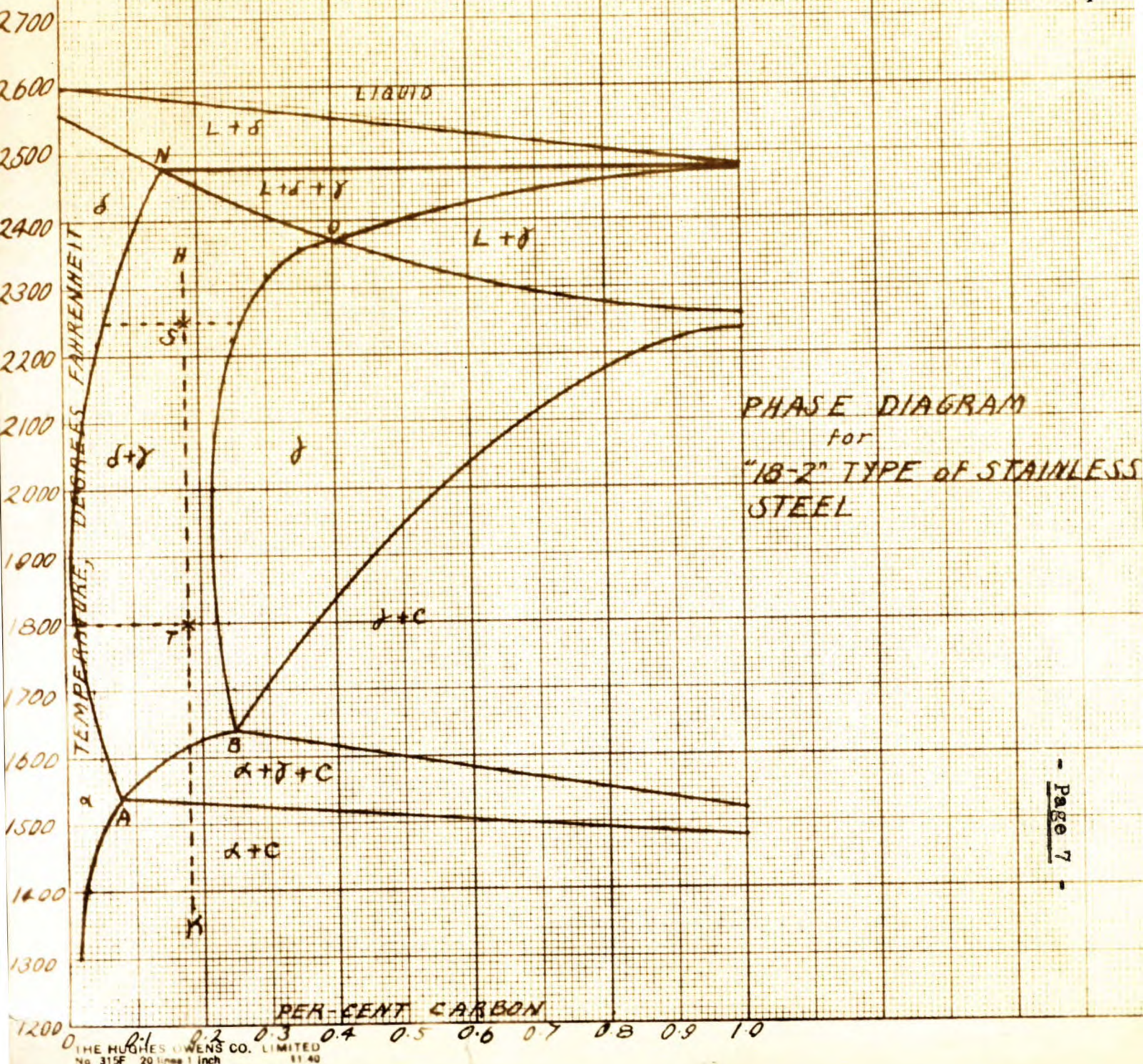
(Microscopic Examination, cont'd) -

Evidence that these carbides are not eliminated after 2 hours at 1800° F. is given in Figure 12. The impact strength after this treatment is 6 foot pounds for A₁ and 4 foot pounds for B₁. This is probably because more carbides were formed in Treatment B than in Treatment A. It will be noted that the martensite in these two specimens etched because they were tempered. The original boundaries of the ferrite that existed after Treatments A and B are still outlined by carbides in Figure 12. Note that some of the ferrite has been converted through the delta-to-gamma reaction to martensite.

Figure 20 shows that even after 10 hours at 1800° F. some of these carbides still persist. The improvement in impact strength is attributed to the elimination of most of this carbide network.

It is evident from Figure 3 that carbides also exist at the martensite-ferrite interface in the steel in the "as received" condition.

(Page 7 is a phase-diagram)
(chart, referred to in the)
("Discussion of Results".)



DISCUSSION OF RESULTS:

To facilitate a discussion of the results of this investigation, a phase diagram for this type of steel is given (Page 7). This is made up from information obtained from the following articles:

"Influence of Nickel on the Chromium-Iron-Carbon Constitutional Diagram," by V. N. Krivobok and M. A. Grossman.

Transactions of American Society for Steel Treating, Volume 18, July-December 1930, page 808.

"A Study of the Iron-Chromium-Carbon Constitutional Diagram," by V. N. Krivobok and M. A. Grossman.

Transactions of American Society for Steel Treating, Volume 18, July-December 1930, page 760.

"Nature of the Chromium-Iron-Carbon Diagram," by Marcus A. Grossman.

Transactions of American Institute of Mining and Metallurgical Engineers, Volume LXXV, 1927, page 214.

It is not held that this diagram is absolutely accurate. However, it is thought to be a close approximation to the phase diagram. For steel in the range being investigated it agrees fairly well with the findings. At all temperatures above, about 1650° F., both delta and gamma iron exist. At 2250° F. more delta iron exists than at 1800° F. This is evident from an examination of Figure 12 and is also evident from the phase diagram. The ratio of the distance between the line HK and OB to the distance between lines NA and OB at any given temperature gives an estimate of the amount of delta iron present at equilibrium. The nearer HK approaches OB the greater will be the amount of gamma phase (or its transformation product) present.

It is therefore evident that the conditions present in the steel satisfy the requirements of the phase diagram

(Discussion of Results, cont'd) -

used to facilitate this discussion.

Line NA represents the solubility of the alpha or delta iron for carbon. It will be noted that this solubility is rather low at 1800° F. but is quite appreciable at 2250° F. According to the phase diagram, delta iron formed at 2250° F. contains about 0.05 per cent carbon. At 1800° F. it can hold only 0.01 per cent carbon in solid solution. The excess will be precipitated as carbide, as is shown in Figures 6 and 10. The slower the cooling the greater is the amount of carbide formed. This is also illustrated in Figures 6 and 10.

Owing to the very slow rate of solution of this carbide at 1800° F., as is evident in Figures 12 and 20, a normal heat treatment could not be expected to remedy this situation.

It is evident, from a comparison of Treatments A₁ and A₃, that this carbide network has an appreciable effect on the impact strength of this steel, as the increased time allowed for solution in Treatment A₃ has raised the impact strength from 6 foot pounds to 11 foot pounds. However, this final value of 11 foot pounds is still far from the original 44.7 foot pounds and in view of the small amount of carbide remaining after Treatment A₃ it is doubtful that complete elimination would restore the original impact strength.

There are two other probable reasons why the impact strength has suffered so drastically from this high-temperature treatment and why further re-heat treatment will not restore it.

The first is the distribution of ferrite. It will be noted that the structure of the steel in its original

(Discussion of Results, cont'd) -

condition consists of long narrow stringers of ferrite and wide stringers of martensite. In this condition the ferrite would have little effect on the impact strength of the steel when the fracture takes place across the direction of rolling. Likewise any free carbides at the ferrite-martensite interface in this type of structure would have little effect on the impact strength of the steel when the fracture takes place across the direction of rolling.

However, when the ferrite is arranged as shown in Figures 14 and 15, the more or less random arrangement of the ferrite might have the effect of quite markedly reducing the impact strength.

Another possibility is that there still persists a carbon concentration in the form of sub-microscopic carbides or a higher-carbon martensitic phase at the grain boundaries and that the rate of solution and diffusion of the carbides is so sluggish that no amount of heat treatment alone can restore the toughness of the metal. Forging would be the only way to break up this network of carbon concentration.

To show what effect ferrite distribution would have on the impact properties, impact bars could be obtained across the direction of rolling and parallel to the direction of rolling of bar stock. If the results obtained vary widely, it would indicate that ferrite distribution has an influence on the impact strength.

To check on the effect of carbon concentration the following experiment is suggested: Obtain a piece of S-80 steel of known impact properties. Heat to 2250° F. and air cool. Re-heat treat a portion of this piece and cut impact

(Discussion of Results, cont'd) -

bars from it. Forge the remainder at 1900° F. to 1700° F. in such a manner as to avoid directional properties. This will thoroughly break up the carbide network, without giving an orderly distribution to the ferrite phase. Heat-treat this forged piece and determine its impact value. If the original impact strength has been completely restored, carbon distribution and not ferrite distribution is the critical factor in the behaviour of this steel. If the original impact strength is still only partly restored, ferrite distribution is also a factor.

CONCLUSIONS:

When "S-80" stainless steel is heated to 2250° F. and slowly cooled (either air-cooled or furnace-cooled), the original impact strength of the steel is destroyed and subsequent heat treatment will not restore it completely.

The reason for this lies in at least two, and possibly all, of the following:

1. A carbide network is formed on cooling from this temperature. This network can only be removed by subsequent soaking at the quenching temperature for from 10 to 20 hours. When this is done the impact strength is somewhat improved.
2. Although the visible carbide network is eliminated there is the possibility that a carbon concentration still persists at the grain boundaries in the martensite and that it is impossible to break this up by heat treatment alone.
3. The more or less random arrangement of the ferrite caused by the high-temperature treatment may be the cause of the poor impact properties.

If either 2 or 3 is correct, only forging at a proper temperature could restore the impact properties. The experiment outlined at the close of the discussion should throw some light on this subject.

BIBLIOGRAPHY:

STAINLESS IRON AND STEEL - J. H. G. Monypenny.
Published by Chapman & Hall, Ltd., London.

CHROMIUM STEELS - Richard Henry Greaves.
Published by His Majesty's Stationery
Office, London.

THE BOOK OF STAINLESS STEELS - Edited by
Ernest E. Thum.
Published by American Society for Metals,
Cleveland, Ohio.

PRACTICAL METALLOGRAPHY OF THE STAINLESS STEELS -
Stanley F. Watkins.
Published in "Metals and Alloys," January,
February, March and October, 1941.

A STUDY OF THE IRON-CHROMIUM-CARBON CONSTITUTIONAL
DIAGRAM - V. N. Krivobok and M. A. Grossman.
American Society for Steel Treating, Vol. 18,
1930, p. 760.

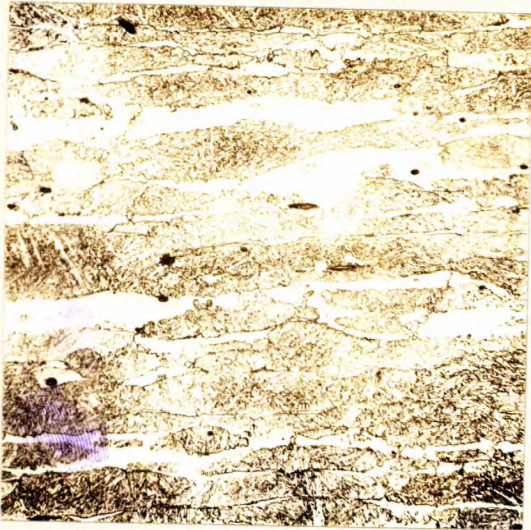
INFLUENCE OF NICKEL ON THE CHROMIUM-IRON-CARBON
CONSTITUTIONAL DIAGRAM - V. N. Krivobok and
M. A. Grossman.
American Society for Steel Treating, Vol. 18,
1930, p. 808.

AN INTRODUCTION TO THE IRON-CHROMIUM-NICKEL ALLOYS -
Edgar C. Bain and William E. Griffith.
Transactions of the American Institute of
Mining and Metallurgical Engineers,
Vol. LXXV, 1927, p. 166.

NATURE OF THE CHROMIUM-IRON-CARBON DIAGRAM -
Marcus A. Grossman.
Transactions of the American Institute of
Mining and Metallurgical Engineers,
Vol. LXXV, 1927, p. 214.

oooooooooooo
oooooo
oo

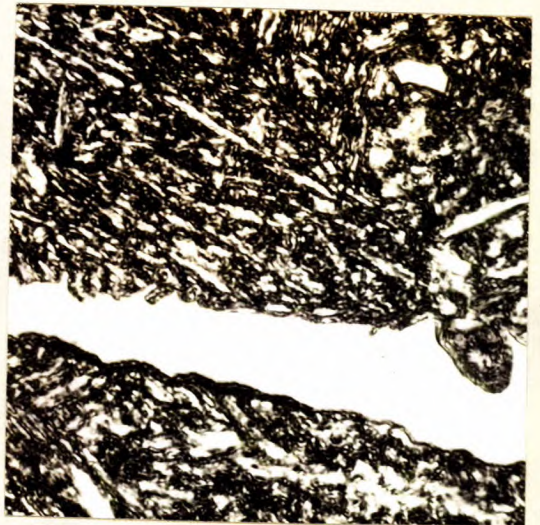
Figure 1.



Photomicrograph, X100.
Electro-polish, etched in 5% HCl
1% picric acid, solution in alcohol.

STRUCTURE AS RECEIVED.

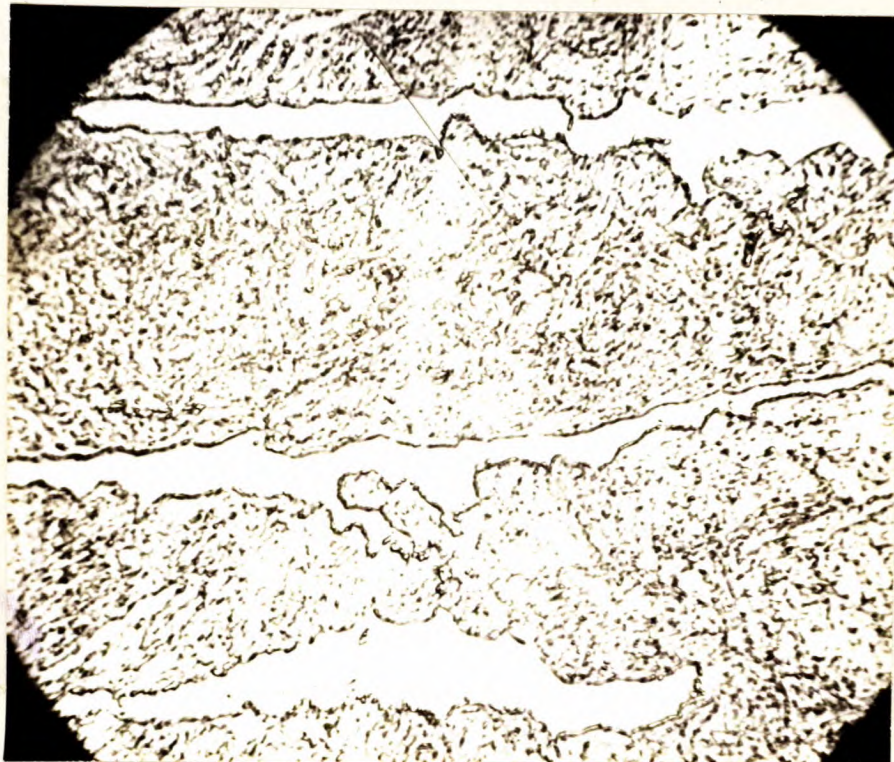
Figure 2.



Photomicrograph, X1000.
Electro-polish, etched in alcoholic
solution of 5% HCl and
1% picric acid.

"AS RECEIVED" CONDITION.

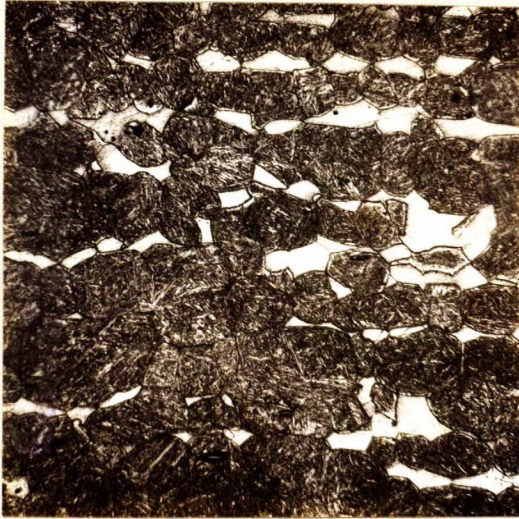
Figure 3.



X1000. Hand polish, electrolytic etch in
10% aqueous tartaric acid solution, 6V, 0.15A, 8 minutes.

STRUCTURE IN STEEL AS RECEIVED.

Figure 4.



X100. Electro-polish,
etch in alcoholic solution
of 5% HCl and 1% picric acid.

STRUCTURE WHEN HEATED TO 2250° F.,
HELD FOR 5 HOURS, AND AIR COOLED.

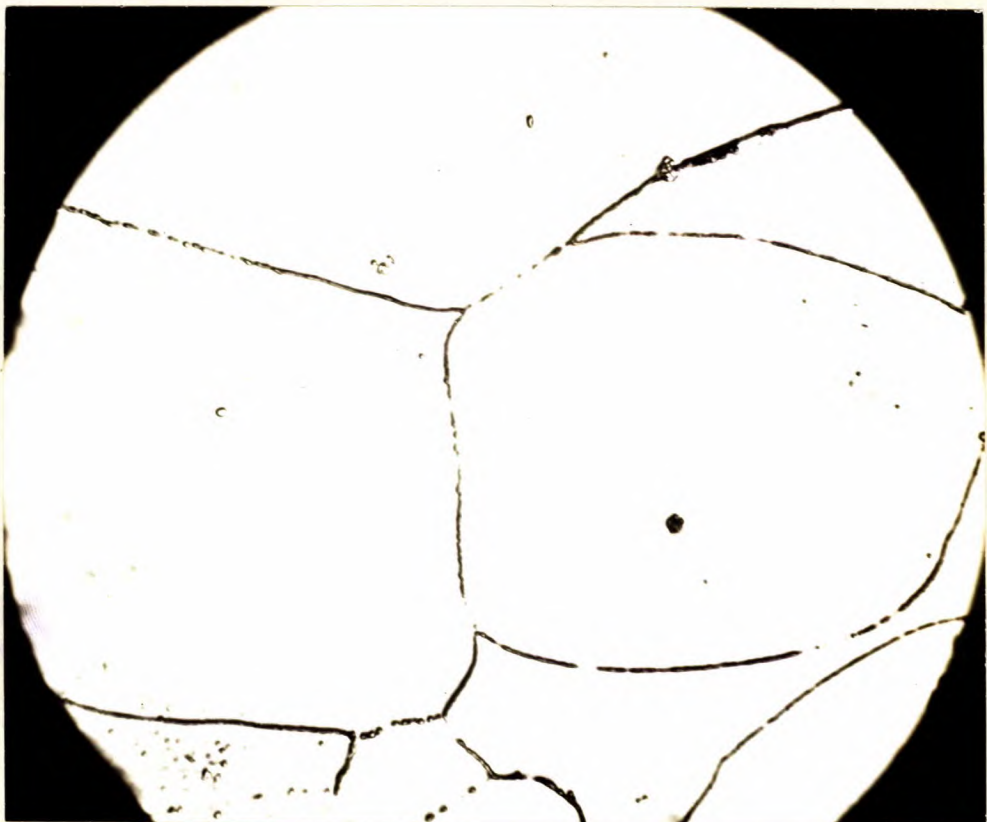
Figure 5.



X1000. Electro-polish,
etch in alcoholic solution
of 5% HCl and 1% picric acid.

STRUCTURE WHEN HEATED TO 2250° F.,
HELD FOR 5 HOURS, AND AIR COOLED.

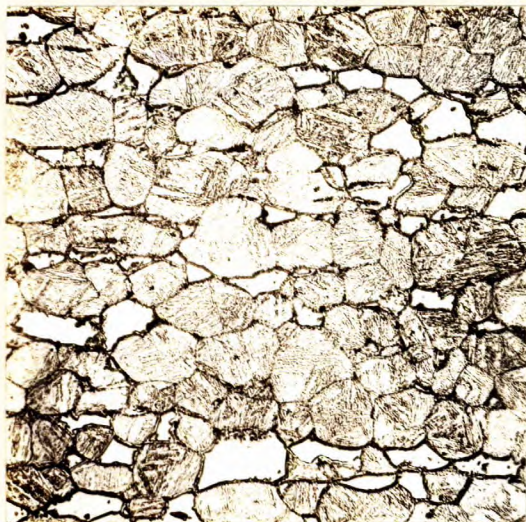
Figure 6.



X1000. Hand polish, electrolytic etch in
10% aqueous tartaric acid solution, 6V, 0.15A, 8 minutes.

CARBIDE NETWORK CREATED BY HEATING TO 2250° F.,
HOLDING FOR 5 HOURS, AND AIR COOLING.

Figure 7.



X100. Electro-polish,
etch in alcoholic solution
of 5% HCl and 1% picric acid.

STRUCTURE WHEN HEATED TO 2250° F.,
HELD FOR 5 HOURS, COOLED TO 1300° F.,
HELD FOR 5 HOURS, AIR COOLED.

Figure 8.



X1000. Electro-polish,
Etch in alcoholic solution
of 5% HCl and 1% picric acid.

STRUCTURE WHEN HEATED TO 2250° F.,
HELD FOR 5 HOURS, COOLED TO 1300° F.,
HELD FOR 5 HOURS, AIR COOLED.

Figure 9.



X2000. Hand polish, electrolytic etch in
10% alcoholic hydrochloric acid solution, 4V, 0.4A, 10 seconds.

STRUCTURE WHEN HEATED TO 2250° F., HELD FOR 5 HOURS,
COOLED TO 1300° F., HELD FOR 5 HOURS, AIR COOLED.

Reveals carbide phase (clear white) around areas of ferrite.

Figure 10.

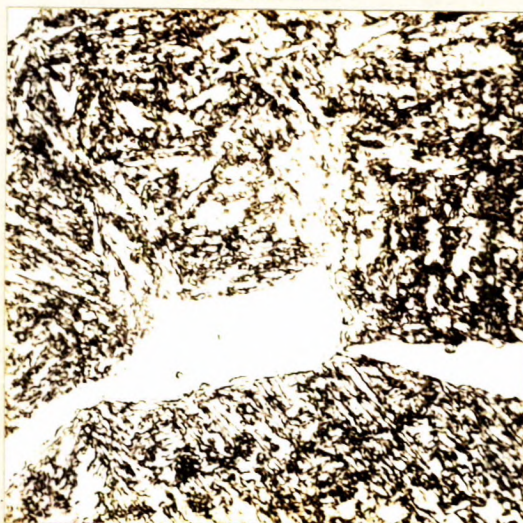


X1000. Hand polish, electrolytic etch in 10% aqueous solution of tartaric acid, 6V, 0.15A, 8 minutes.

STRUCTURE WHEN HEATED TO 2250° F., HELD FOR 5 HOURS, COOLED TO 1300° F., HELD FOR 5 HOURS, AIR COOLED.

Note carbide at grain boundaries and also in ferrite. Large grains are martensite.

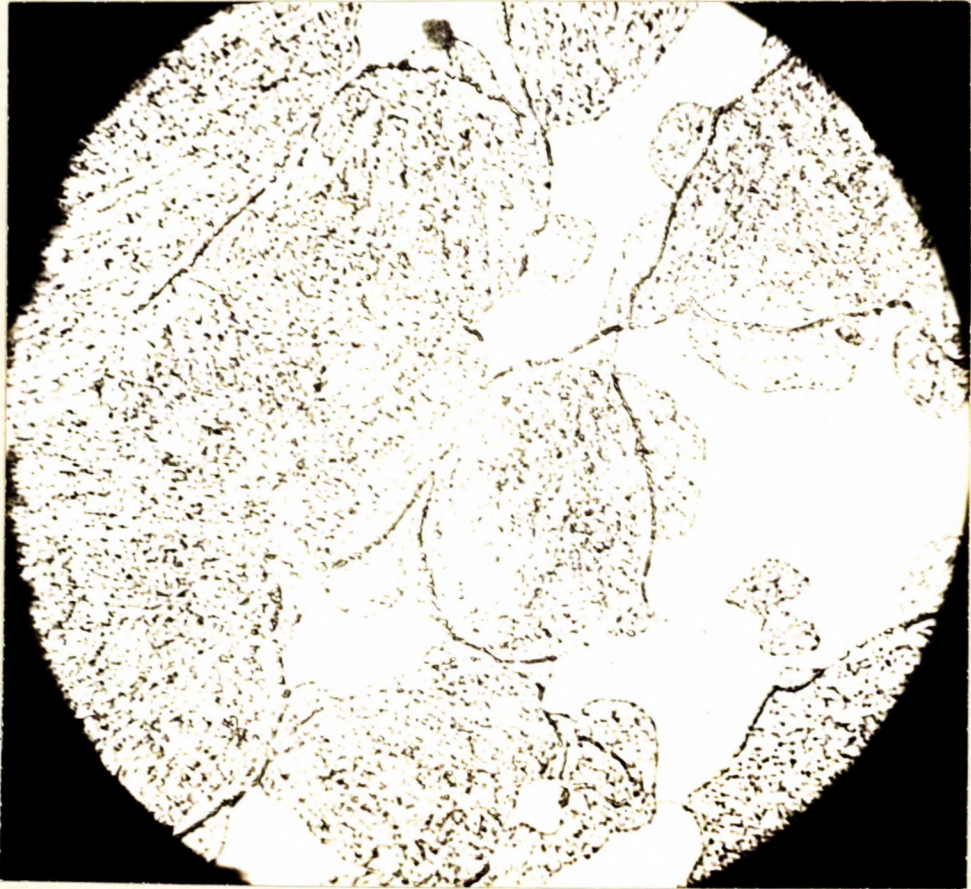
Figure 11.



X1000. Electro-polish, etch in alcoholic solution of 5% HCl, 1% picric acid.

STRUCTURE WHEN HEATED TO 2250° F., HELD FOR 5 HOURS, AIR COOLED; HEATED TO 1800° F., HELD FOR 2 HOURS, OIL QUENCHED; AND HEATED TO 1200° F., HELD FOR 1 HOUR, OIL QUENCHED.

Figure 12.



X1000. Hand polish, electrolytic etch in 10% aqueous tartaric acid solution, 6V, 0.15A, 8 minutes.

STRUCTURE WHEN HELD AT 2250° F. FOR 5 HOURS,
AIR COOLED; HEATED TO 1800° F., HELD FOR 2 HOURS
OIL QUENCHED; AND HEATED TO 1200° F., HELD FOR
1 HOUR, OIL QUENCHED.

Note persistence of carbide network formed by 2250° F. treatment. This exists at the old boundary between ferrite and martensite. Note that amount of ferrite has decreased.

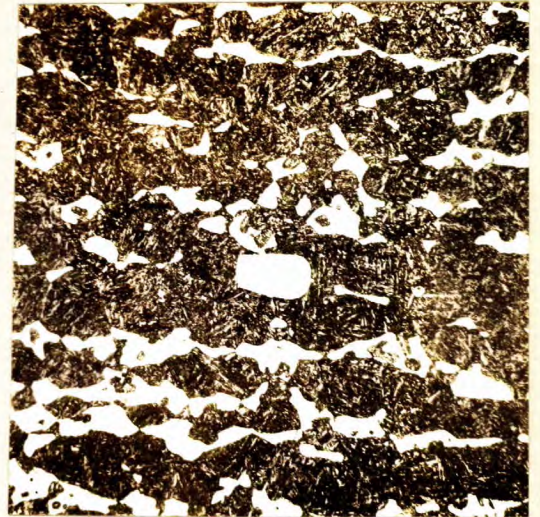
Figure 13.



X1000. Electro-polish, etch
in alcoholic solution of 5% HCl,
1% picric acid.

STRUCTURE WHEN HEATED TO 2250° F.,
HELD FOR 5 HOURS, COOLED TO 1300° F.,
HELD FOR 5 HOURS, AIR COOLED; HEATED
TO 1800° F., HELD FOR 2 HOURS, OIL
QUENCHED; HEATED TO 1200° F., HELD
FOR 1 HOUR, OIL QUENCHED.

Figure 14.



X100. Electro-polish, etch
in alcoholic solution of 5% HCl
and 1% picric acid.

STRUCTURE WHEN HEATED TO 2250° F.,
HELD FOR 5 HOURS, AIR COOLED;
HEATED TO 1850° F., HELD FOR
3 HOURS, AIR COOLED; HEATED TO
1800° F., HELD FOR 2 HOURS, OIL
QUENCHED; HEATED TO 1200° F.,
HELD FOR 1 HOUR, OIL QUENCHED.

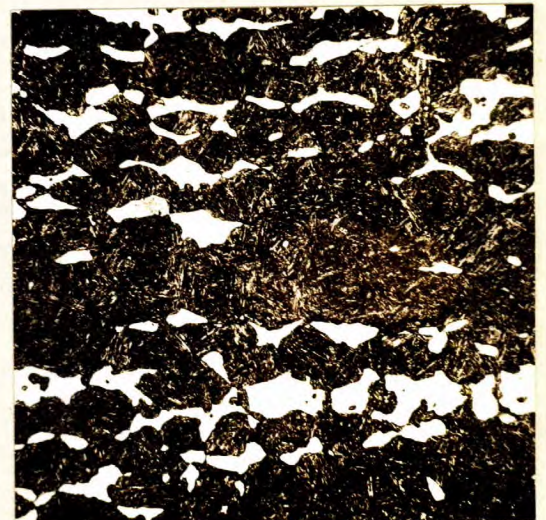
Figure 15.



Photomicrograph, X1000.

SAME AS FIGURE 14.

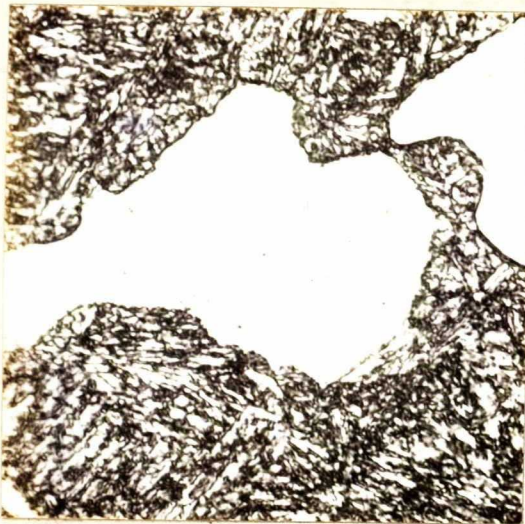
Figure 16.



X100. Electro-polish, etch
in alcoholic solution of 5% HCl
and 1% picric acid.

STRUCTURE AFTER HEATING TO 2250° F.,
HELD 5 HOURS, COOL TO 1300° F., HELD 5
HOURS, AIR COOLED; HEATED TO 1850° F.,
HELD 3 HOURS, A.C., HEATED TO 1800° F.,
HELD 2 HOURS, O.Q.; AND HEATED TO
1200° F., HELD 1 HOUR, O.Q.

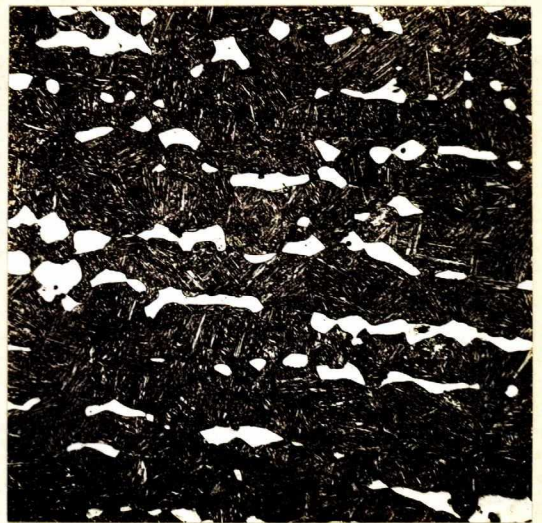
Figure 17.



Photomicrograph, X1000.

SAME AS FIGURE 16.

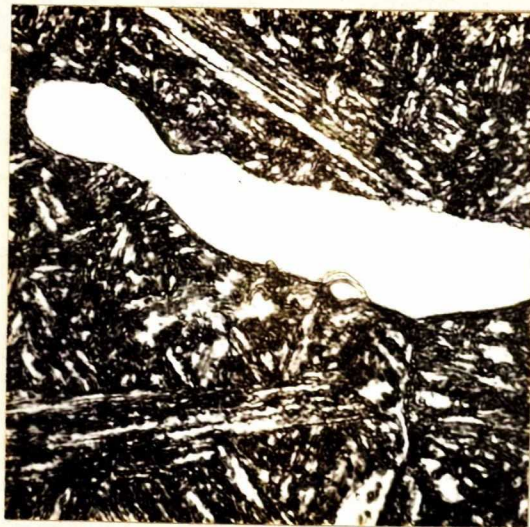
Figure 18.



X100. Electro-polish, etch
in alcoholic solution of 10% HCl
and 1% picric acid.

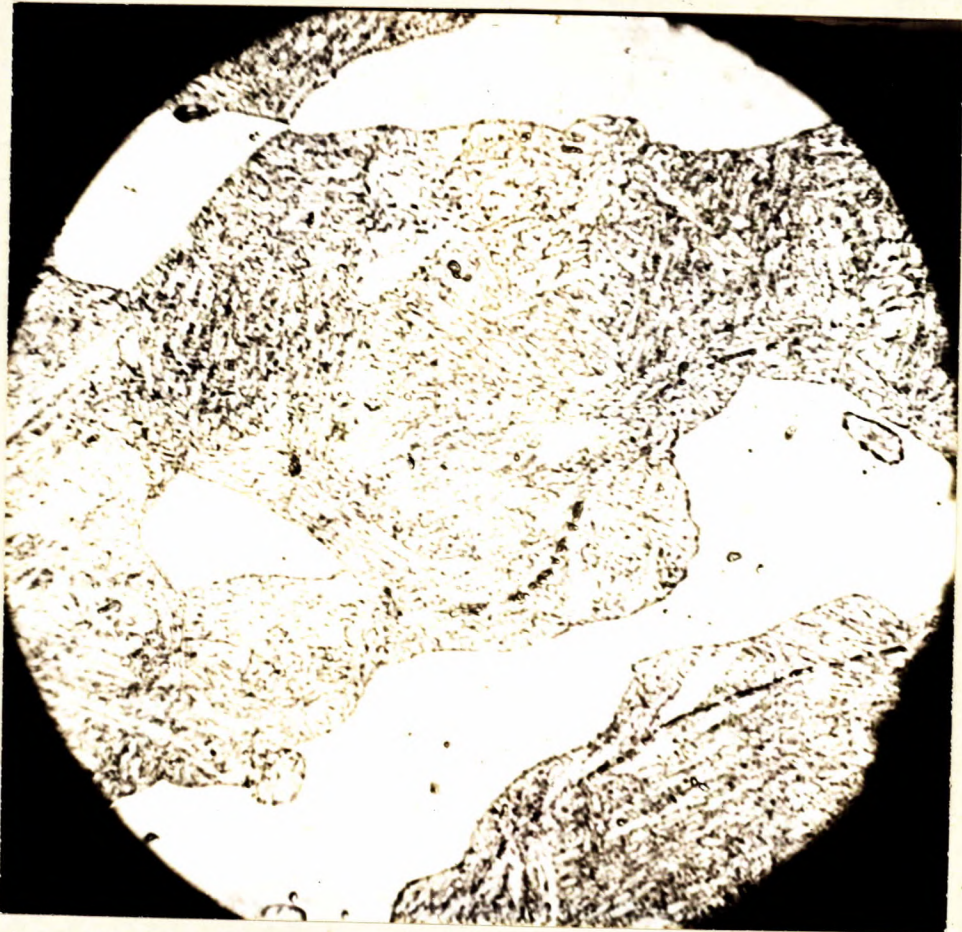
STRUCTURE AFTER HEATING TO 2250° F
5 HOURS, AIR COOLED; 1800° F.,
10 HOURS, O.Q.; 1200° F., 1 HOUR,
O.Q.

Figure 19.



X1000. Same as Figure 18, except
electrolytically etched in 10%
alcoholic solution of HCl for
10 seconds at 4V and 0.4A.

Figure 20.



X1000. Hand polish, electrolytic etch in 10% aqueous tartaric acid solution, 6V, 0.15A, 8 minutes.

STRUCTURE AFTER HEATING TO 2250° F.,
5 HOURS AND AIR COOLED; 1800° F., 10 HOURS,
OIL QUENCH; 1200° F., 1 HOUR, OIL QUENCHED.

Note that much of the carbide network has been
dissolved by the 10-hour treatment at 1800° F.

HVK:GHB.