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METALLURGICAL EXAMINATION OF ABEE METEORITIC IRON

by

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METALLURGICAL EXAMINATION OF ABEE METEORITIC IRON

Chemical analysis of iron extracted from the Abee meteorite by an iodide-methanol reflux method showed the average nickel content of the iron to be of the order of 6.2% and calculations indicate that with perfect separation the nickel analysis will be increased to about 7%. The combined carbon content of the iron is at least 0.19% and the total carbon content of the bulk meteorite is at least 0.89%.

The iron shows evidence of reheating and is believed to be a low-nickel ataxite, although, depending on the interpretation of a microscopic, hard acicular phase (alpha - 2), the meteorite might be assigned to the coarsest octahedrite class. No Neumann lines were observed in the iron.

The iron in this meteorite generally responds to quenching, tempering and spheroidizing heat treatment like an 0.45% carbon steel except that the pearlite ferrite structure has a hardness of R_C 40 and is more resistant to spheroidization than normal. The development of a granular enveloping (bainite-plessite) phase at the surface of ferrite grains on annealing from 1600°F probably indicates marginal concentration of the nickel at the surface of the ferrite.

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INTRODUCTION

On November 16, 1959, Dr. K. R. Dawson of the Geological Survey of Canada Branch, Petrology Section, submitted three polished petrographic samples from the Abee meteorite (containing a mixture of metallic and non-metallic constituents) with the request that the Physical Metallurgy Division examine the iron fraction of this meteorite. Two additional samples were submitted a week later, a sample $1 \times \frac{1}{4} \times \frac{1}{4}$ in. taken from the fusion crust, and a block $l\frac{1}{2} \times \frac{3}{4} \times \frac{1}{2}$ in. cut from the interior of the meteorite.

Previous work (1), done to obtain the bulk analysis of the whole meteorite, without separation of the metallic and nonmetallic fractions, indicated that the nickel content and total carbon content were of the order of 1.6% and 1.5% respectively. It was hoped to establish the distribution of nickel and carbon between the metallic and non-metallic fractions of the meteorite minerals and thereby assist in classification of the meteorite. Identification of a phase, present in the ferrite (kamacite) which resembled pearlite was also requested.

As shown in Figures 1 and 2 the meteorite consisted of such small fragments of non-metallic and metallic constituents that mechanical separation was impractical and chemical separation (2) of the iron was required.

An attempt was made to separate the metallic from the nonmetallic portion of the Abee meteorite using an iodide-methanol reflux reaction. This separation yielded a soluble portion of $44.29 \stackrel{+}{=} 0.92\%$ and a nickel content of $4.53 \stackrel{+}{=} 0.07\%$ on this portion.

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Subsequent improvement in separation increased the nickel analysis to 6.2% and calculations indicate that with complete separation an average nickel analysis of the order of 7% will be obtained.

SCOPE AND OBJECTS OF INVESTIGATION

The scope of this metallurgical examination was limited to examination of the metallic portion of the meteorite with the following objects:-

- (1) determination of the nickel, cobalt, manganese, phosphorous,
 total and combined carbon contents of the iron after
 separation.
- (2) study of the phases present in the metallics and detection of the presence of Neumann bands; of graphite; of vestigial traces of Widmanstatten structure or of the Alpha-2 phase reported in previous investigations.
- (3) study of the etching response to nital, picral and alkaline sodium picrate solutions.
- (4) measurement, by microhardness tests, of the hardness of phases ^x present.
- (5) determining the response of the meteorite iron to conventional heat treatments (for iron and steel of "artificial" origin) such as:- full-annealing; spheroidizing; hardening and tempering.
- (6) comparison of the extent of surface oxidation present on samples heat treated in vacuum or air with that present on the fusion crust of the meteorite.

In addition to tests on the phases present in the metallics some comparative hardness tests were made on prominent mineral phases.

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IDENTIFICATION AND DISPOSITION OF MATERIAL

The samples examined and the disposition of these samples are listed in Table 1.

TABLE 1.

IDENTIFICATION OF SAMPLES

Sample "No.	Disposition
63	x microexamination
68	microexamination and heat treatment
4	microexamination and heat treatment
13 - 2	microexamination, microhardness tests, chemical analysis and heat treatment
1-1	microexamination and microhardness tests on the fusion crust
x	

All samples were examined as polished and after etching 15 seconds in 4% picral solution. The samples were then repolished and were etched in boiling alkaline sodium picrate solution for $l_{\overline{z}}^{1}$ minutes (The latter etchant removed sulphide mineral and attacked Fe₃C).

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HEAT TREATMENT TESTS

Two specimens of Sample 13 - 2 approximately $\frac{1}{4}$ in. diameter and $\frac{3}{4}$ in.length were sealed in evacuated silica tubes for hardening and annealing heat treatments from 1550°F and 1600°F respectively.

The first sample was heated (in vacuum) to 1550°F, was held at 1550°F for 20 minutes followed by breaking of the tube and quenching of the sample to 60°F in cold 10% brine. This sample (13-2-WQ-vac) was examined to observe any change in hardness or microstructure. The quenched sample was then reheated to 300°F for 2 hours in air followed by air-cooling (13-2-WQ-300°F). Half of this specimen was again reheated in air to 1250°F for 1 hour and was allowed to cool in air to room temperature (13-2-WQ-1250°F).

The second sample of 13-2, sealed in an evacuated silica tube, was heated to 1600°F, was held 30 minutes at 1600°F and was then allowed to cool (in vacuum) in the furnace to room temperature after which the tube was broken and the specimen was examined for changes in hardness and microstructure (13-2-FC-vac). This test was duplicated on a 3/8" cube cut from sample No.4, except that heating was carried out in air to allow decarburization of the surface (4-FC -air). After heat treatment the cube was halved and the cross section was examined to determine if changes in microstructure, carbon content and hardness had occurred.

Half of Sample 68 was heated at $1100^{\circ}F$ for 120 hours in air in an attempt to spheroidize the lamellar constituent present in the ferrite (68-1100°F.)

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The heat treatment tests and identification code letters are summarized in Table 2.

TABLE 2.

SUMMARY OF HEAT TREATMENT TESTS

Lett	er Sample	Heat Treatment
A	(13-2-WQ-vac)	x Sealed in vacuum 1550°F - 20 minutes. WQ to room temperature.
в	(13 - 2-WQ-300°F)	Half of first sample tempered 2 hours at 300°F in air.
C	(13-2-WQ-1250°F)	Half of Sample B additionally tempered 1 hour at 1250°F in air.
D	(13-2-FC-vac)	Sealed in vacuum 1600°F - 30 minutes, cooled in furnace to room temperature.
Е	(4-FC-air)	Same as D except that sample was not held in vacuum.
F	(68-1100°F)	3/8" cube of sample 68 was heated 120 hours in air at 1100°F.

WQ - water-quenched in 10% brine at 60°F.

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RESULTS

The iodide-methanol reflux separation of the metallic from the non-metallic portion of the meteorite yielded a soluble portion of 44.29 \pm 0.92% (6 determinations). The analysis of the soluble portion is shown in Table 3.

TABLE 3

ANALYSIS OF THE IODIDE-METHANOL SOLUBLE PORTION EXTRACTED

FROM THE ABEE METEORITE (44.29 \pm 0.92% SOLUBLE PORTION) (2)

0.19 ± 0.05 ^X	2
72.19 ± 0.16	3
4.53 ± 0.07	2
0.25 ± 0.01	2
0.37 - 0.01	2
13.54 ^X	1.
0.80 ^X	l
	0.19 ± 0.05^{X} 72.19 ± 0.16 4.53 ± 0.07 0.25 ± 0.01 0.37 ± 0.01 13.54^{X} 0.80^{X}

These values are obtained by calculations using the difference between the total ion concentration in the bulk sample and the quantity of this ion which was found in the iodine-methanol insoluble portion.

An improved separation which reduced the quantity of sulphur, from the sulphide minerals, in the soluble portion gave an average nickel content of 6.2% and indicated that with complete separation an average nickel content of the order of 7 per cent would be obtained in the iron fraction of the meteorite.

Microhardness results obtained on samples 13-2 and 1-1 are shown in Table 4 for the phases observed in the metallic fraction and for a few of the prominent minerals. Results are included for constituents present in samples which were heat treated in laboratory furnaces.

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TABLE 4.

RESULTS OF MICROHARDNESS TESTS

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Sample No.	X _{Re} Description of Phase	ockwell C Hardness
<u></u>	As Received Meteoritic Iron	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,
13-2	alpha iron (ferrite or kamacite)	35 to 44
11	alpha iron and Fe ₃ C (pearlite)	42 to 48
It	"alpha 2" (3) (martensite or plessite)	58 to 63
11	alpha 2 - granular (bainite or plessite)	57
H	light grey sulphide phase) impression	
n	dark grey sulphide phase)adjacent mineral	s 47 (combined
11	dark grey glassy silicate	cracked or poor impression
18	light grey "enstatite"needles	67 to 70
1-1	oxide silicate fusion crust (magnetite)	39 to 59
n	decarburized alpha iron	37 to 38
11	"alpha 2"(3)	56
11	martensite (near surface and slightly tempered)	58 to 61
11	ferritic areas	38
lt	pearlite areas	41 to 43
	Heat Treated Laboratory Specimens	
A	martensite	60
В	slightly tempered martensite	62
C	martensite tempered 2 hours at 300°F + 1 hour at 1250°F	42 to 45
D	enveloping "bainite" or "plessite"	56 to 58
IT	pearlite	41
Е	enveloping "bainite" or "plessite"	56

Sample No.	Description of Phase	Rockwell C Hardness
E	pearlite	44 to 47
F	decarburized areas of alpha iron	33
n	alpha iron which was not decarburized	38
. 11	pearlite - partly dissolved	44 to 45
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(100g load, 10.25 objective. Tukon microhardness tester)

The hardness in Rockwell C was obtained by conversion from Knoop hardness and was checked against Rc64; Rc47 and Rc27 standard calibration blocks. The letters A-F inclusive designate samples which were given the laboratory heat treatments shown in Table 2.

METALLOGRAPHIC OBSERVATIONS (AS-RECEIVED SAMPLES)

Examination of the specimens in the as-polished condition showed the ferrite as white areas intimately associated with a light grey and a beige-coloured phase. The grey and beige phases are believed to be sulphide minerals (CaS and FeS). Also prominent in all photomicrographs are a light grey and a dark grey silicate phase. The light grey silicate phase (enstatite) is commonly present in and adjacent to the ferrite. No Neumann lines were observed in any of the numerous ferrite areas examined. Typical areas containing ferrite and silicate mineral are illustrated at 100 diameters in Figures 1 and 2.

The sulphide minerals are more plainly visible in Figure 3 at higher magnification.

Figures 4, 5 and 6 illustrate various minerals present in the ferrite which were observed on the as-polished surface before etching. These constituents were not identified.

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Figures 7, 8, 9 and 10 illustrate silicate mineral closely bonded to the ferrite. The acicular hard mineral illustrated in Figures 9 and 10 has been termed "enstatite" throughout this report.

Figures 11 and 12 illustrate two ferrite areas which have been etched 15 seconds in 4% picral solution. The iron appears "veined" and contains a lamellar phase resembling the pearlite^X phase of "artificial" iron and steel. Numerous small inclusions may be phosphide mineral (rhabdites) which have been commonly observed in meteoritic irons.

Deeper etching in picral resulted in heavier attack of the lamellar constituent and revealed evidence of chemical segregation (coring) in the ferrite and traces of retained austenite (taenite) as shown in Figures 13 and 14.

The appearance of typical ferrite pearlite areas, after etching l_2^1 minutes in boiling alkaline sodium picrate, is illustrated in Figures 15 and 16. The severe etch has removed the sulphide minerals and has attacked the lamellar phase so that the lamellae appear black when viewed at low magnification. This response on etching in boiling alkaline sodium picrate is characteristic of the behaviour of Fe₃C in "artificial" steels, tending to confirm that the phases shown are ferrite (kamacite) and Fe₃C (cementite or cohenite).

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Pearlite is the entectoid transformation product of gamma iron obtained by cooling at rates below the critical cooling rate in "artificial" irons and steels which contain carbon.

Small areas, present at the surface of ferrite particles or present as projections at the surface of the ferrite were hard and were believed to be bainite (plessite) resulting from the transformation of nickel-rich austenite (taenite) which was present at the mineral interface of some ferrite particles. The presence of this bainite phase (Figures 17 and 18) at the surface of the ferrite suggested the marginal concentration of the nickel responsible for the 4.5% - 7% average composition. Occasionally small islands or parts of a discontinuous envelope of retained austenite (taenite) were observed at the surface of the ferrite, however, taenite was present only in trace amounts. The lamellar phase in the ferrite, shown in Figures 17 and 18 is believed to be Fe₃ C similar to that shown previously in Figures 15 and 16, however it was not established whether the lamellar phase is discontinuous with the bainite (plessite) envelope.

The appearance of small islands of an acicular phase sometimes present at the extremities of ferrite particles and sometimes as small grains divorced from the larger ferrite grains is illustrated in Figures 19, 20, 21 and 22. This constituent is the hardest metallic constituent observed in the as-received samples and is believed to be a form of lower bainite, (alpha 2 phase) but might also be interpreted as vestigial Widmanstatten except for the fact that it has a hardness up to $R_c 63$.

AS-RECEIVED FUSION CRUST SAMPLE 1-1

Figures 23 and 24 illustrate the structures observed in small areas of surface metal which were melted during passage through the

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earth's atmosphere. The phases illustrated in Figure 23 are believed to be delta iron, martensite and retained austenite or ferrite. In Figure 24 the microstructure is similar but a larger martensite area is visible.

Figure 25 illustrates the appearance of the fusion crust in Sample 1-1 in the as-polished condition. Figure 26 illustrates a similar crust area after etching for $l_{\overline{z}}^1$ minutes in boiling alkaline sodium picrate. An adjacent area contained numerous particles of iron which had been transformed to martensite during atmospheric flight. Figure 27 illustrates an area about $\frac{1}{4}$ inch from the crust surface with the ferrite-carbide structure etched 40 seconds in 4% picral.

SAMPLES HEAT TREATED IN THE LABORATORY

Figure 28 illustrates the appearance of Sample B, which was water-quenched from 1550°F and tempered 2 hours at 300°F. The hardness and microstructure resemble slightly tempered martensite. A carbon content of the order of 0.45% and a severe quench would be required to produce this microstructure in carbon steel.

Figure 29 illustrates the appearance of Sample D, furnacecooled from 1600°F in vacuum. The lamellar phase is evident. There was also a change observed at the surface of the ferrite so that larger quantities of the bainite (plessite) enveloping phase are observed in this sample than in the as-received samples. The appearance of this phase on annealing suggests the presence of a marginal concentration of the nickel in the ferrite or as taenite. (In an artificial steel, heating to 1600°F should produce

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homogeneous austenite and rapid diffusion of carbon so that a uniform distribution of pearlite in ferrite would result from slow cooling in the absence of alloy segregation).

Figures 30 and 31 illustrate Sample E, etched 30 seconds in nital or $l_{\overline{z}}^{1}$ minutes in boiling alkaline sodium picrate respectively. Some of the exposed ferrite has been partially decarburized by heating in an oxidizing atmosphere for $\frac{1}{\overline{z}}$ hour at 1600°F. A layer of oxide, caused by the furnace treatment, is visible at the top of each photomicrograph. However, after furnace-cooling pearlite is present in all the ferrite areas which were not directly exposed to the oxidizing furnace atmosphere. (One bainite area was observed which appeared to be inside a ferrite area but this location for bainite was exceptional.)

Figures 32, 33, 34 and 35 illustrate the appearance of Sample F, heated at 1100°F for 120 hours. Partial solution and spheroidization of the carbide is demonstrated. In some ferrite particles, exposed to the oxidizing atmosphere during heating, a distinct diffusion band (Nickel-Oxygen) was observed at the particle surface and the carbide phase was almost completely absent. In this condition the minimum hardness of R_c 33 was obtained.

Figure 36 illustrates the microstructure observed in Sample B, water-quenched and tempered at 300°F at higher magnification than was previously illustrated in Figure 28.

Figure 37 illustrates more advanced decomposition of the martensite to ferrite and carbide in sample C, tempered 1 hour at 1250°F.

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SUMMARY OF OBSERVATIONS

- (1) No Neumann bands were observed in the ferrite.
- (2) No graphite was observed in the metallic phase with the possible exception of traces in areas such as were illustrated in Figures 4, 5 and 6.
- (3) The metallic iron particles contain an average content of 4.5% nickel and a minimum combined carbon content of 0.19%. Calculations indicate that with more complete separation of the iron the nickel analysis will be increased to about 7% and the combined carbon content will also increase.
- (4) The lamellar phase, commonly present in all the ferrite particles, resembles Fe3 C in its response to the boiling alkaline sodium picrate etch, and by the fact that at 1550°F it was dissolved and was retained in solution by water quenching to form a hard phase (martensite). The martensite phase responded to stress-relieving at 300°F and to tempering at 1250°F. Frolonged heating at 1100°F (120 hours) resulted in partial spheroidization of these lamellae. Annealing, by heating at 1600°F and cooling at about 100°F/hour produced a microstructure similar to the "as-received" meteorite except for development of an envelope of granular plessite or bainite at the surface of some of the ferrite particles. The "as-quenched" hardness of the meteoritic iron corresponded to that carbon steel having a minimum carbon content of 0.45%. The slight increase in hardness, from R $_{\rm c}60$ to R $_{\rm c}63$ after tempering at 300°F may signify transformation of some austenite

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retained during quenching.

- (5) The lamellar phase, present in the as-received samples or developed in the furnace-annealed test sample, resembled Fe₃C (cohenite) in appearance but in distribution was coarser and more irregular than the symmetrical random distribution characteristic of the pearlite observed in artificial steels containing carbon. The ferrite-pearlite structure of the meteoritic iron at R_c 38 was considerably harder than that observed in artificial steels and homogenization of the iron at 1600°F was more difficult than normal. Spheroidization at 1100°F was also slower than normal.
- (6) In the as-received meteorite, traces of taenite and its decomposition product (plessite) were observed as thin films on the surface or as surface projections on some ferrite particles. The traces of retained austenite (nickel-rich taenite) remained white while the ferrite was darkened by the picral etch. In the furnace-cooled laboratory samples, however, the taenite appeared to be transformed to plessite or bainite and the thickness of the envelope around ferrite grains was increased, possibly by diffusion of marginally concentrated nickel inwards in ferrite grains.
- (7) An acicular phase resembling the alpha-2 phase described by H. H. Uhlig⁽³⁾, was observed in the as-received meteoritic iron. This phase was widely distributed in the as-received samples but occurred in small quantities only visible at a magnification of the order of 500 diameters. The phase was

- 14 - 1

often completely divorced from ferrite grains but was also present at ferrite surfaces where projections extended to the silicate mineral. This phase was the hardest metallic phase observed and seemed to be aform of martensite or lower bainite which differed from the granular enveloping bainite slightly in hardness and by having a well-developed acicular structure. (Except for its high hardness this phase might be identified as a microscopic form of vestigial Widmanstatten structure).

Examination of the fusion crust, sample 1-1, showed a (8) relatively hard light grey oxide believed to be magnetite. Some of the ferrite close to the crust appeared to be decarburized. A zone of martensite was observed in the fusion crust beneath the decarburized crust which resembled the martensite developed by water-quenching from 1550°F. This martensite zone resulted from heating during flight in the earth's atmosphere followed by rapid cooling of the hot crust by the cold subsurface material. At the extreme crust surface a few small fragments, Figures 23 and 24, were observed which had been molten during flight and which contained delta iron, martensite and retained austenite or ferrite. The depth of the fusion crust and heated zone was about 1/8 in. and below this zone the characteristic structure of ferrite and lamellar carbide was observed.

CONCLUSIONS

- The meteoritic iron exhibits no Neumann lines and contains
 6 7% nickel and a minimum of 0.19% combined carbon. The iron contains sufficient combined carbon to form martensite when the metal is brine-quenched from 1550°F.
- (2) The lamellar phase is believed to be cohenite (Fe₃C) but is somewhat harder (R_c 38) than artificial ferrite-pearlite structures and appeared to resist spheroidization to the extent that while partial solution and partial spheroidization occurred, some unspheroidized lamellae remained after 120 hours at 1100°F.
- (3) An acicular phase, having a hardness of R_c 63, was identified as martensite (alpha 2) phase, possibly resulting from the transformation of nickel-rich areas of retained austenite. A granular bainitic phase having a slightly lower hardness than martensite, R_c 58, was observed in the as-received meteorite and, in much larger quantities, as an envelope at the surface of ferrite grains in the laboratory furnace-annealed sample. The increased amount of the phase after heating to 1600°F and furnace-cooling may be explainable by diffusion of marginally concentrated nickel into the ferrite grains.
- (4) The fact that no Neumann lines were observed, that the ferrite had penetrated and filled fine cracks in the enstatite and the globular appearance of some ferrite grains suggested that this meteorite was altered from its original form. While

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segregation of the nickel to the iron surface suggested extremely slow cooling under equilibrium conditions, the fact that the lamellar phase was not spheroidized also indicated reheating and cooling of the meteorite at a rate faster than the equilibrium rate.

(5) The meteorite could tentatively be classified as a lownickel ataxite (altered hexahedrite) although it might possibly fit in the group of coarsest octahedrites, depending on the origin of the alpha 2 phase.

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(X100)





Figure 2 - Sample 13-2. As-Polished.

Intimate mixture of meteoritic iron and silicate mineral which necessitated chemical extraction of the iron for analysis.



(X500)

Figure 3 - 13-2 As-Polished.

Sulphide minerals are visible at the ferritesilicate interface.



(X500)

Figure 4 - 13-2. As-Polished.

Note the ferrite filling minute cracks in the enstatite.

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(X500)



Unidentified phases visible in the as-polished condition.



(X500)

Figure 6 -13-2 As-Polished.

Unidentified phases visible in the as-polished condition.



(X500)

Figure 7 - 13-2 As-Polished

Enstatite, sulphide mineral and alpha iron.



(X500)

Figure 8 - 13-2

As-Polished

Sulphide mineral is visible at the interface between the iron and the silicate mineral.



(X500)

Figure 9 - As-Polished.

Enstatite crystals (silicate mineral associated with the iron)



(X500)

Figure 10 - As-Polished.

Enstatite and traces of unidentified grain boundary constituent.



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(X500)

Figure 11 Sample 13-2 Etched 15 seconds in 4% Picral.

The iron contains a lamellar phase and numerous small inclusions which are probably rhabdites (phosphides).



Figure 12 Sample 13-2 Etched 15 seconds in 4% Picral.

The response to etching in picral is similar to that of an artificial steel and reveals a lamellar phase in the ferrite.





Islands of taenite (white constituent) are visible at the left of this photomicrograph.



(X500)

Figure 14. Sample 13-2 Etched 1 minute in 4% Picral.

This photomicrograph illustrates a carbide intergrowth in the ferrite. The grey phase at the surface of the ferrite is a sulphide phase. Some of the white phase at the surface of the ferrite may be taenite but it is believed to be mainly a second sulphide phase.



(X750)

Figure 15. Sample 13-2 Etched 1¹/₂ minutes in boiling alkaline sodium picrate.

This etchant has attacked and darkened the carbide intergrowth.



Figure 16. Sample 13-2 - Etched 12 minutes in boiling alkaline sodium picrate.

This photomicrograph illustrates "pearlite" in a ferrite grain. The hardness of this metallic area is R $_{\rm C}$ 38.



(X500)



Traces of a partly granular bainitic (plessite) phase are visible at projections and at the surface of the ferrite.





Sample 13-2 Etched l_{2}^{1} minutes in boiling Figure 18. alkaline sodium picrate.

> This photomicrograph illustrates two ferrite grains having envelopes of a bainitic (plessite) phase. The hardness of the ferrite-pearlite area is about R_{c} 40; of the bainitic phase is about R_{c} 57.



(X750)

Figure 19. Sample 13-2 Etched 12 minutes in boiling alkaline sodium picrate.

Alpha 2 phase, believed to be lower bainite or martensite, having a hardness of R $_{\rm C}$ 63.



(X750)

Figure 20. Sample 13-2 Etched 1¹/₂ minutes in boiling alkaline sodium picrate.

This photomicrograph illustrates a small grain which is half ferrite and half alpha 2 phase.



(X500)

Figure 21. Sample 13-2 Etched l_{Ξ}^{1} minutes in boiling alkaline sodium picrate.

This photomicrograph illustrates an area where several small grains of the alpha 2 phase are associated with ferrite-pearlite grains.





Figure 22. Sample 13-2 Etched 1 minutes in boiling alkaline sodium picrate.

This photomicrograph illustrates the alpha 2 phase present at projections of a ferrite grain.



Figure 23 Sample 1-1. Extreme Crust Surface. Etched 4% Picral.

Metal which has been molten on the fusion crust surface. The phases are believed to be delta iron, martensite, and either ferrite or retained austenite.



Figure 24

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Sample 1-1 Extreme Crust Surface. Etched 4% Picral.

An area similar to that of Figure 23 showing martensite needles.





An oxidized layer (magnetite) is visible at the surface of this sample.



(X500)

Figure 26. Sample 1-1 Etched l_{z}^{1} minutes in boiling alkaline sodium picrate.

Same area as Figure 25 at higher magnification. Areas of tempered martensite were observed adjacent to this area.



(X500)

Figure 27. Ferrite-Pearlite Area Inside Crust Sample 1-1. Etched 40 seconds 4% picral.



(X500)

Figure 28. Sample B Etched 1¹/₂ minutes in Boiling Alkaline Sodium Picrate.

Quenched and slightly tempered martensite Rc63.



(X500)

Figure 29. Sample D . Etched $l_2^{\frac{1}{2}}$ minutes in Boiling Alkaline Sodium Picrate.

Furnace cooled from 1600°F. The annealed structure consists of ferrite-pearlite areas often surrounded by a bainitic (plessite) envelope.



(X150)

Figure 30. Sample E 30 seconds nital etch.



(X150)

Figure 31. Sample E. 12 minutes boiling alkaline sodium picrate etch.

Same area as Figure 25.

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Figure 32. Sample F. 30 seconds 4% picral

Illustrates some spheroidization and small islands which may be taenite or traces of the sulphide phase.



Figure 33. Sample F. 30 seconds 4% picral

Heating at 1100°F for 120 hours has resulted in considerable but not complete spheroidization of the lamellae.



Figure 34. Sample F. $\frac{1}{2}$ minute in boiling alkaline sodium picrate.

Partial solution and spheroidization are illustrated.



Figure 35. Sample F. 1 minute in boiling alkaline sodium picrate.

Spheroidization is more advanced in the grain at the top of this picture than in the other grain. In this condition the hardness varied between R_c 33 and R_c 45 depending on the quantity of carbide present.



(X500)

Figure 36. Sample B $\frac{1}{2}$ minute in boiling alkaline sodium picrate.

Brine-quenched. Tempered 2 hours at 300°F. Slightly Tempered Martensite Rc 62.



(X500)

Figure 37. Sample C $\frac{1}{2}$ minute in boiling alkaline sodium picrate.

Same as the sample shown in Figure 36 - tempered at 1250°F. The martensite has decomposed to carbide and ferrite having a hardness of Rc 42-45.