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REPORT

of the

ORE DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 1443.

Progress Report on Investigation into Factors Affecting Life of Cast Steel Retorts as Used by Dominion Magnesium Limited.

(Copy No. 10.)

Abstract

This interim report lists the result of examination made of material used in high alloy cast steel retorts employed in the ferro-silicon reduction process for magnesium.

Neither variations in density nor room temperature mechanical properties were found to bear any relationship to retort life. In comparing chemical analysis obtained after service with that reported by the foundry, varying degrees of decarburization were noted. However, this did not appear to bear any relationship with retort life.

Microscopic examination indicated that the presence of massive carbides, probably originating in the cast structure, might have some bearing on retort performance. These carbides became soluble at 2300° F. and showed a tendency to precipitate again at 2100° F. The investigation is proceeding and as sufficient additional information is obtained, further reports will be issued.

Bureau of Mines
Division of Metallic
Minerals
-
Ore Dressing
and Metallurgical
Laboratories

CANADA

DEPARTMENT
OF
MINES AND RESOURCES

Mines and Geology Branch

O T T A W A   July 12th, 1943.

[redacted]

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Investigation No. 1443.

Progress Report on Investigation into Factors
Affecting Life of Cast Steel Retorts as
Used by Dominion Magnesium Limited.

Material Submitted and Object of Investigation:

On May 26th, 1943, samples from six cast alloy steel retorts were submitted for examination by Mr. C. K. Lockwood, Asst. Sales Manager, Shawinigan Chemicals Limited, Montreal, Quebec. Five of these samples were taken from retorts that had been in service at the Dominion Magnesium Limited's magnesium reduction plant at Haley's Station, Ontario. One sample was taken from a similar type of retort which had not been in service. Service records of the used samples were markedly different.

The object of this work was to determine whether or not there was any difference metallurgically among the

(Material Submitted and Object of Investigation, cont'd) -

samples submitted which might account for the variations in service performance.

This report is of an interim nature and any conclusions reached therein should be regarded as being tentative only.

Table I gives a list of the samples received.

Table I. - Samples Received.

Sample Retort No.	Remarks
2728-B	5/8-in. wall, long life. Selected as representative of a thin-walled retort giving good life.
2877-B-2	Short life. 60 days to first blow-up. 110 days total life.
2905-B-2	5/8-in. wall, 95 days total life. Selected as representative of a thin-walled retort giving short life.
2802-B-1	Given five blow-ups in service. Selected to determine whether or not blow-ups have any affect on structure.
2832-B-2	This retort was reported to have given exceptionally long life. 215 days total life with two blow-ups; 74 days between blow-ups.
3162-B-1	This retort had never seen service. It was selected to function as an 'as cast' sample.

As pointed out in our Report of Investigation No. 1415, "Life of Stainless Steel Retorts: A Survey of Records up to May 12th, 1943," (May 26th, 1943), none of the recorded factors, such as chemical analysis, wall thickness, furnace used, position in furnace, etc., appears to have any effect on retort life. There are indications that some variables in foundry practice, such as condition of moulding sand and core sand, method of gating and risering, design of mould, pouring rate, casting temperature, etc., probably have a greater effect

(Material Submitted and Object of Investigation, cont'd) -

on the retort life than metal quality. However, as the centrifugal casting of these retorts, a procedure that should largely eliminate casting unsoundness, is planned, any information that could be obtained on the effect of metal quality would probably be of value in the planning of future operations.

Density Determinations:

Large samples, about 4" x 8" in size, were used for density determinations. These determinations were made by weighing in air and in water. The results are recorded in Table II.

Table II. - Density Determinations.

Retort No.	Density, grams per cubic centimeter	Life of Retort
2728-B	7.958	5/8-in. wall, long life.
2877-B-2	7.959	60 days to first blow-up, 110 days total.
2905-B-2	7.895	5/8-in. wall, 95 days total life.
2802-B-1	7.966	5 blow-ups, long life.
2832-B-2	7.873	215 days total life.
3162-B-1	7.950	New retort.

Mechanical Properties:

Standard tensile bars were prepared from each retort. The results obtained are recorded in Table III.

Table III. - MECHANICAL PROPERTIES.

Retort No.	Ultimate tensile strength, p.s.i.	Yield strength, p.s.i.	Elongation in 2 inches, per cent	Reduction of area, per cent	Hardness, Rockwell 'B'
2728-B	60,500	35,000	6	8	64
2877-B-2	58,000	31,000	18.5	19	69
2905-B-2	66,000	32,000	21	26.5	64
2802-B-1	57,000	29,500	32.5	42	60
2832-B-2	Flaw in bar.*		-	-	65
3162-B-1	41,500	31,000	8	26	68

* Test not repeated because of shortage of material.

Chemical Analysis:

The chemical analyses obtained on the retort samples as received here are compared, in Table IV, below, with those reported by the foundry casting the retorts.

Table IV. - Chemical Analysis.

		Sample Retort No.				
	2728-B	2877-B-2	2905-B-2	2802-B-1	2832-B-2	3162-B-1
Carbon, per cent						
O.D.M.L. -	0.28	0.24	0.11	0.03	0.07	0.12
Foundry -	0.35	0.27	0.22	0.19	0.29	
Silicon, per cent						
O.D.M.L. -	0.78	0.61	0.69	0.52	0.81	1.24
Foundry -	0.67	0.52	0.65	0.42	0.84	
Nickel, per cent						
O.D.M.L. -	35.11	37.42	35.37	36.43	33.75	38.16
Foundry -	34.65	36.90	35.10	35.60	32.65	
Chromium, per cent						
O.D.M.L. -	16.76	17.83	16.39	16.38	16.55	15.84
Foundry -	16.69	18.04	16.73	16.91	16.40	
Manganese, per cent						
O.D.M.L. -	1.02	0.94	0.88	1.08	1.12	0.41
Sulphur, per cent						
O.D.M.L. -	0.020	0.022	0.020	0.022	0.018	0.012
Phosphorus, per cent						
O.D.M.L. -	0.019	0.029	0.020	0.015	0.021	0.020
Molybdenum, per cent						
O.D.M.L. -	0.02	0.02	0.02	0.03	0.10	0.13

It will be noted that in Retorts Nos. 2728-B and 2832-B-2 both gave over 200 days' life and yet Retort No. 2832-B only lost 0.07 per cent of carbon while Retort No. 2832-B-2 lost 0.22 per cent of carbon.

Microscopic Examination:

Samples from each retort were prepared for the microscope. There was evidence of intergranular microscopic shrinkage in all samples. Characteristic examples of this are shown in Figures 1, 2, 3 and 4.

For structural examination, either electrolytic

(Microscopic Examination, cont'd) -

etching in 10 per cent oxalic acid, or a solution of 10 grams ferric chloride, 30 c.c. hydrochloric acid, and 120 c.c. water, was used. All polishing was done by hand. Table V gives a list of the photomicrographs of the structures in the "as received" condition.

TABLE V. - PHOTOMICROGRAPHS OF "AS RECEIVED" STRUCTURES.

Figure No.	Retort No.	Magnification	Etch
5	2728-B	X25	$FeCl_3$-HCl soln.
6	2728-B	X100	$FeCl_3$-HCl soln.
7	2877-B-2	X25	$FeCl_3$-HCl soln.
8	2877-B-2	X100	$FeCl_3$-HCl soln.
9	2905-B-2	X25	$FeCl_3$-HCl soln.
10	2905-B-2	X100	$FeCl_3$-HCl soln.
11	2802-B-1	X25	Electrolytic etch.
12	2802-B-1	X100	Electrolytic etch.
13	2832-B-2	X25	$FeCl_3$-HCl soln.
14	2832-B-2	X100	$FeCl_3$-HCl soln.
15	3162-B-1	X25	Electrolytic etch.
16	3162-B-1	X100	Electrolytic etch.

Heat-Treatment Tests:

A series of heat-treatment experiments were conducted to determine whether or not it was possible for carbides to exist in the steel at the operating temperature of the retort which is 1150° C. (2100° F. approx.). Table VI gives a list of the various heat treatments tried and the resulting structures as recorded in photomicrographs.

(Continued on next page)

(Heat Treatment Tests, cont'd) -

TABLE VI. - HEAT-TREATMENT EXPERIMENTS.

Retort No.	Temperature, °F.	Time at Temperature, in hours	Method of Cooling	Figure No.	Magnification
3162-B-1	2000	10	W.Q.	17	X25
	2100	20	W.Q.	18	X25
	2200	10	W.Q.	19	X25
	2300	10	W.Q.	20	X25
2877-B-2	2100	20	W.Q.	21	X100
	2200	10	W.Q.	22	X100
	2300	10	W.Q.	23	X100
2905-B-2	2300	10	W.Q.	24	X100
2728-B	2300	10	W.Q.	25	X100
3162-B-1	2300	10	W.Q.)	26	X100
	2100	10	W.Q.)		
2905-B-2	2300	10	W.Q.)	27	X100
	2100	10	W.Q.)		
2728-B-2	2300	10	W.Q.	28	X100
	2100	10	W.Q.	29	X1000
2877-B-2	2300	10	W.Q.	30	X100
	2100	10	W.Q.	31	X1000

The following facts will be noted from an examination of the photomicrographs, Figures 17 to 31:

1. In the case of the 'as cast' sample, Retort No. 3162-B-1, a temperature of 2300° F. was required to effectively disperse the 'as cast' structure. (See Figures 17 to 20).

2. After 20 hours at 2100° F., the carbides appearing in Retorts Nos. 2877-B-2, 2905-B-2, and 2728-B are still present. (See Figure 21).

3. At 2300° F., these carbides dissolve and may be retained in solution by water quenching. (See Figures 23, 24, and 25).

4. On subsequent reheating to 2100° F., carbides are precipitated. The sample which contained the most carbides before heat treatment (Retort No. 2877-B-2), precipitated the most upon reheating to 2100° F. after a solution treatment at 2300° F. (See Figures 26 to 31).

Discussion:

The slight variations in density apparently bore no relationship to the life of the retorts. However, this is not surprising. A localized segregation of shrinkage in an otherwise sound retort could easily cause premature collapse of the retort.

The mechanical properties of the 'as received' materials, as determined at room temperature, show no relationship to retort life. It is, however, interesting to note that the mechanical properties of the steel from Retort No. 2877-B-2, which had a structure high in carbides and a carbon content of 0.24 per cent, were not appreciably different from those of the steel from Retort No. 2802-B-1, which had a structure free from carbides and a carbon content of 0.03 per cent. Evidently the presence of carbides, when these are distributed as in the steel under investigation, has no effect on the static mechanical properties.

The chemical analysis reveals marked differences of retort decarburization. These differences probably indicate that the atmosphere is not constant in all furnace positions. However, conditions favouring extreme decarburization evidently have no influence on the life of the retort. Retorts Nos. 2832-B-2 and 2802-B-1, which were both severely decarburized, and Retort No. 2728-B, which was lightly decarburized, all gave long life, while Retort No. 2877-B-2, which was lightly decarburized, and Retort No. 2905-B-2, which was fairly heavily decarburized, both gave a short life.

Microscopic examination revealed that there was microscopic shrinkage present in all retorts so that this condition, provided that it is uniformly distributed, evidently does not seriously impair the life of the retort.

It will be noted that the two retorts giving the

(Discussion, cont'd) -

shortest life exhibit a structure containing a large quantity of fairly massive carbides (see Figures 8 and 10). Retort No. 2877-B-2 (Figure 8), which only gave a total life of 110 days, has a structure containing many more carbides than does that of Retort No. 2728-B, which gave a much longer life; this, in spite of the fact that the carbon, both in the 'as cast' analysis and in the analysis after service, was higher in Retort No. 2728-B. This fact seems to indicate that the presence or absence of carbides might have some influence on the performance of the retort and that the carbon content of the steel is not the only factor controlling the amount of carbides present.

It may also be noted, particularly from Figure 7, that these carbides are arranged in a dendritic pattern suggestive of an 'as cast' structure. This could indicate that some factor, such as freezing rate--which could be governed by moulding sand condition, pouring rate, and casting temperature and which might influence the amount of carbides present in the 'as cast' structure--could have some influence on the life of the retorts.

The heat treatment tests have revealed interesting facts. First, it has been shown that the 'as cast' structure of Retort No. 3162-B will not diffuse in a reasonable length of time until a temperature of 2300° F. is reached. Even after 20 hours at 2100° F., there is still a trace of this structure. Secondly, in the case of structures consisting of massive carbides, such as is illustrated by Figure 8 (Retort No. 2877-B-2), these carbides are not soluble until a temperature of 2300° F. is reached. Lastly, after dissolving these carbides by heating to 2300° F., carbides can be caused to re-precipitate, (Discussion, cont'd) -

in some cases at least, by heating to 2100° F.

The fact that carbides precipitate at 2100° F. after being put into solution at a higher temperature, indicates that those carbides present in the retort samples as received very likely were formed upon casting and have never been dissolved. Comparing Retorts Nos. 2728-B and 2877-B-2 in the light of this fact, it would appear that the metal in Retort No. 2728-B, although higher in carbon, has less tendency to form carbides than the metal in Retort No. 2877-B-2. As further evidence of this, when samples of these retorts were given solution treatments at 2300° F., followed by precipitation treatments at 2100° F., the amount of carbides precipitated in Retort No. 2728-B was much less than in Retort No. 2877-B-2.

It will be noted that following a solution treatment at 2300° F. and a precipitation treatment at 2100° F., no carbides were precipitated in Retort No. 3162-B, which had a carbon content of 0.12 per cent, while some carbides were precipitated in Retort No. 2905-B-2, which had a carbon content of 0.11 per cent. Also, it would be reasonable to assume that fewer carbides would be precipitated in Retort No. 2905-B-2 than in Retort No. 2877-B-2 because its carbon content is lower, but when Retort No. 2728-B precipitates even fewer carbides, in spite of its higher carbon content, some factor other than carbon content must be sought. This, again, is evidence that for metals of about the same nominal analysis, some have a greater tendency to form carbides than others.

CONCLUSIONS:

This report is but an interim report, describing the work done to date and commenting on the significance of the findings. Such conclusions as are set forth below are merely tentative pending developments from experiments now in progress or planned for the near future:

1. Microstructures characteristic of good and bad retorts have been identified. Retorts giving long life are characterized by low carbide content.

2. Atmospheric conditions in the furnace apparently vary considerably, since there is a wide variance in the degree of decarburization.

3. The degree of decarburization appears to have no influence on retort life.

4. Carbides present in retort metal appear to have persisted from the 'as cast' structure.

5. The metal in some retorts appears to have a tendency to produce more carbides than the metal in other retorts. Such metal is characteristic of poor retorts. There appears to be some factor other than carbon content influencing this behaviour.

Program for Continued Work:

A type of creep test, using a cantilever beam test bar, is being developed. Tests will be conducted on bars cut from samples on hand and, if possible, on samples quenched from 2300° F. It is also proposed to make up small heats of the metal, in an effort to discover the factor influencing carbide formation and its effect on the creep properties of the metal.

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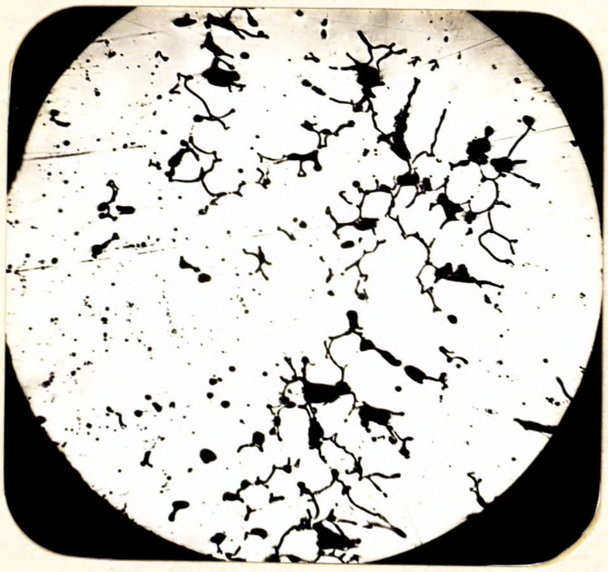
Figure 1.



X25.
RETORT NO. 3162-B-1.

Figure 2.

X100.
RETORT NO. 2832-B-2.

Figure 3.

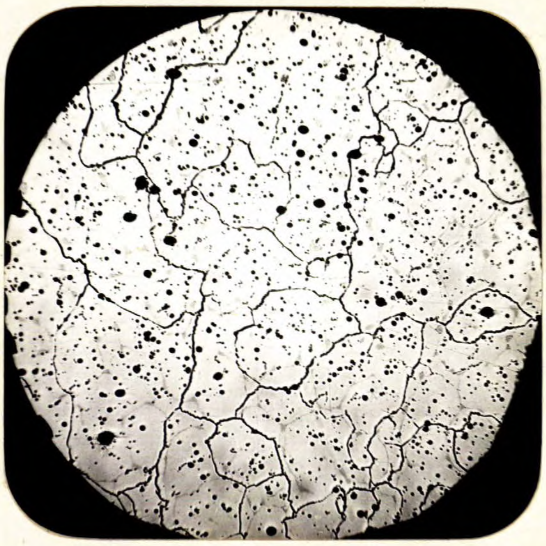
X100.
RETORT NO. 2905-B-2.

Figure 4.

X100.
RETORT NO. 2802-B-1.

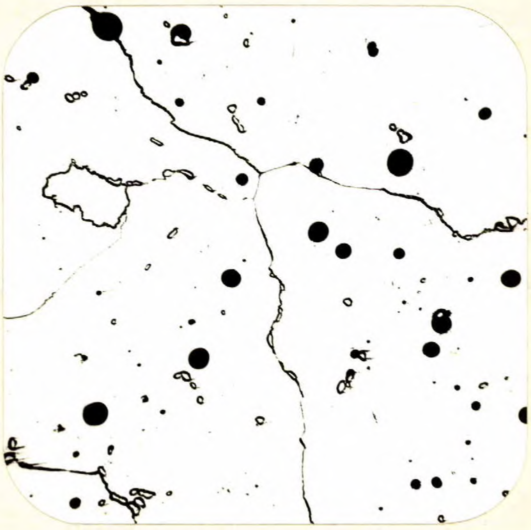
PHOTOMICROGRAPHS TAKEN UNETCHED TO SHOW
TYPICAL EXAMPLES OF INTERCRYSTALLINE SHRINKAGE.

Figure 5.



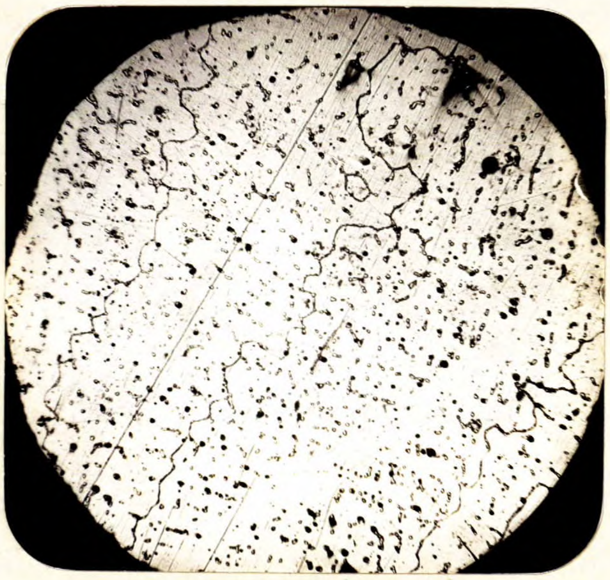
X25.
RETORT NO. 2728-B.
Note grain size.

Figure 6.



X100.
RETORT NO. 2728-B.
Note carbides present in small quantity.

Figure 7.



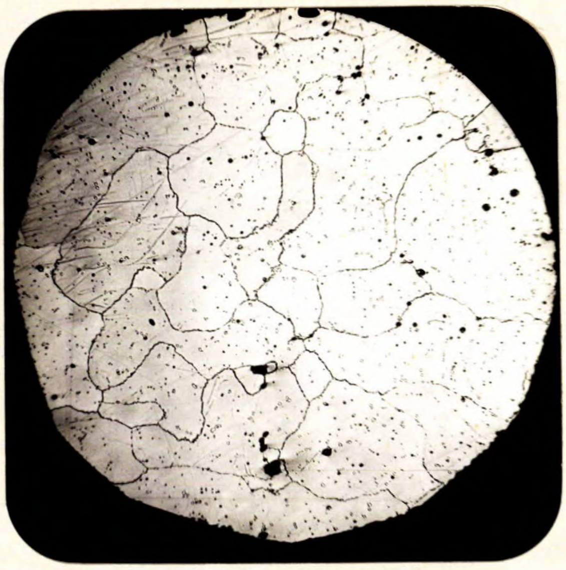
X25.
RETORT NO. 2877-B-2.
Note grain size and dendritic arrangement of carbides.

Figure 8.

X100.
RETORT NO. 2877-B-2.
Note carbides present in large quantities. Compare with Figure 10.

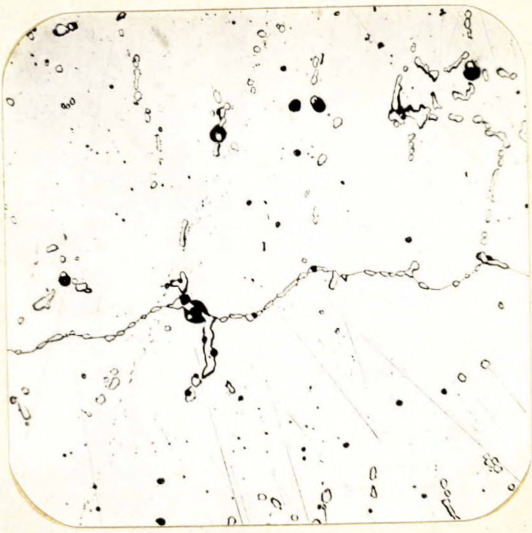
PHOTOMICROGRAPHS OF STRUCTURE AS RECEIVED AFTER SERVICE.

Figure 9.



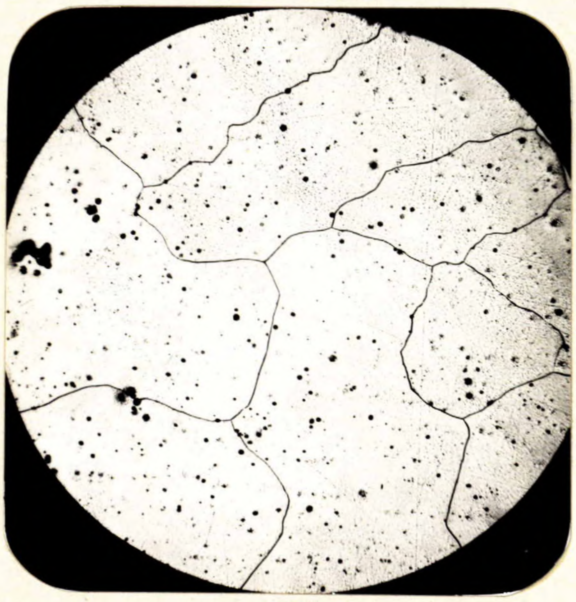
X25.
RETORT NO. 2905-B-2.
Note grain size.

Figure 10.



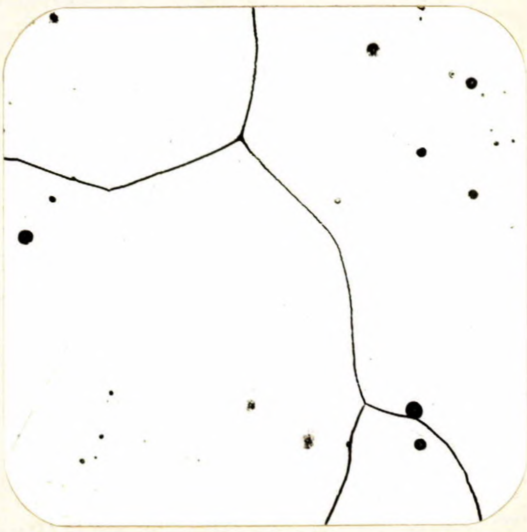
X100.
RETORT NO. 2905-B-2.
Note presence of carbides.
Compare with Figures 7 and 8.

Figure 11.



X25.
RETORT NO. 2802-B-1.
Note grain size.

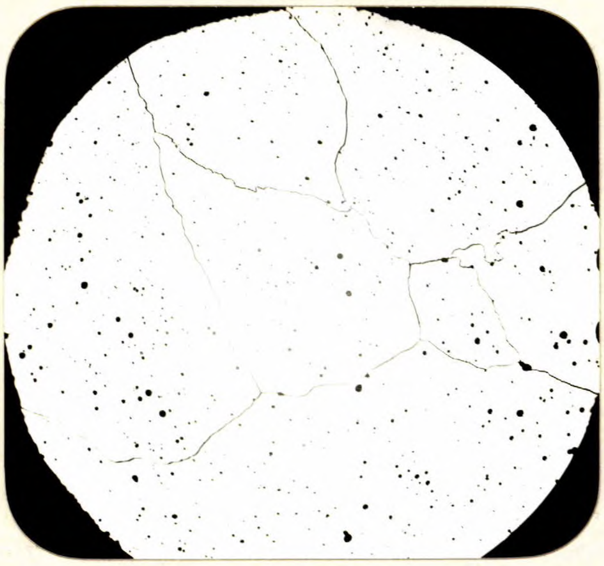
Figure 12.



X100.
RETORT NO. 2802-B-1.
Note absence of carbides.

PHOTOMICROGRAPHS OF STRUCTURE
AS RECEIVED AFTER SERVICE.

Figure 13.

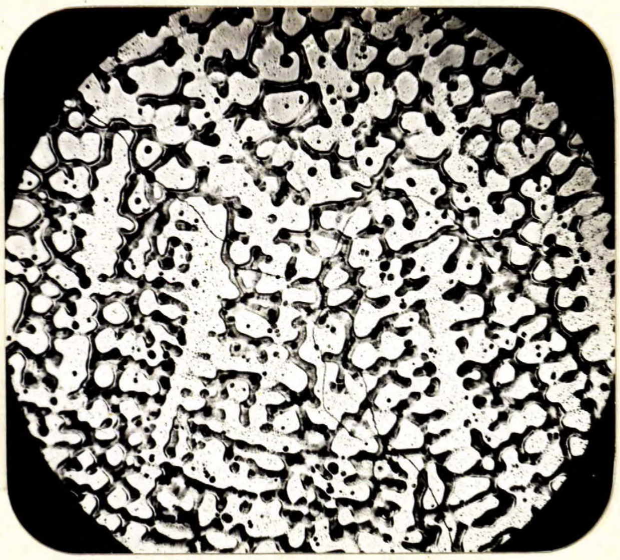


X25.
RETORT NO. 2832-B-2.
Note grain size.

Figure 14.

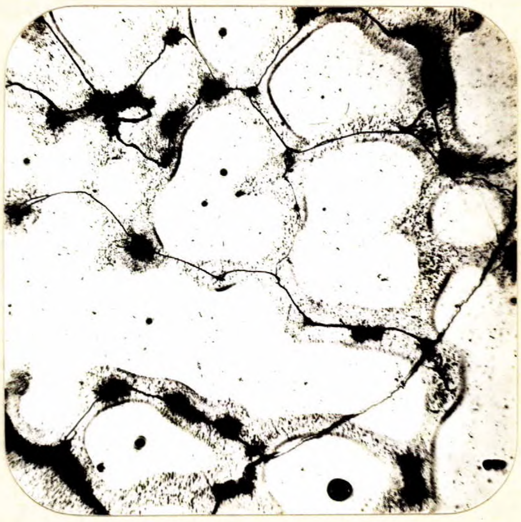
X100.
RETORT NO. 2832-B-2.
Note absence of carbides.

Figure 15.



X25.
RETORT NO. 3162-B-1.
'As cast' structure. Note segregated network.

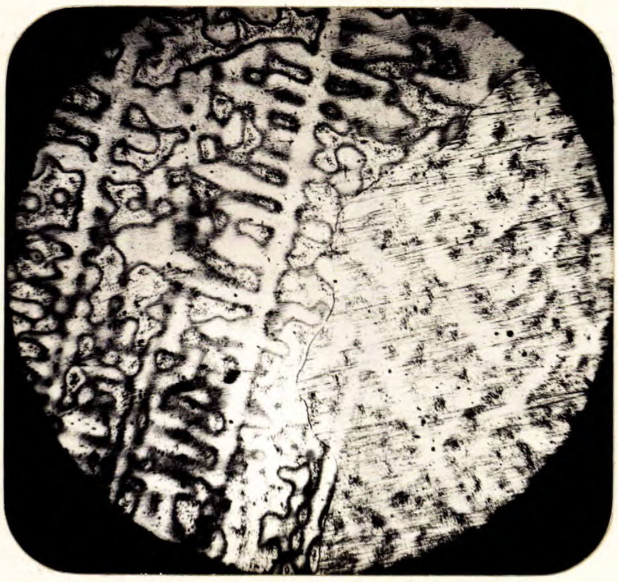
Figure 16.



X100.
RETORT NO. 3162-B-1.
'As cast' structure.

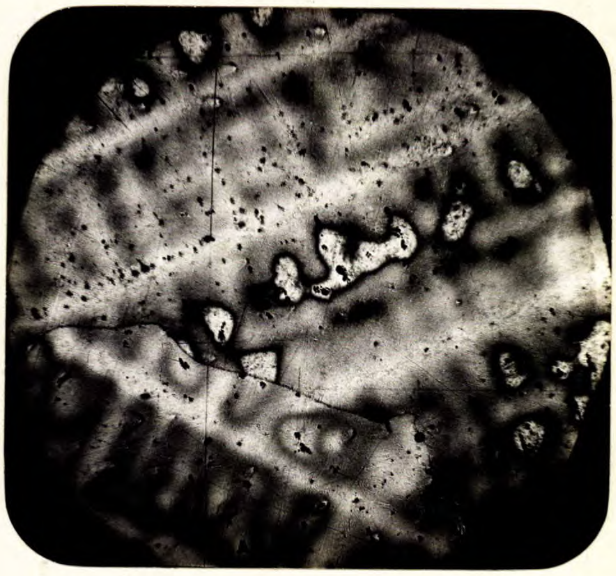
PHOTOMICROGRAPHS OF STRUCTURE
AS RECEIVED AFTER SERVICE.

Figure 17.



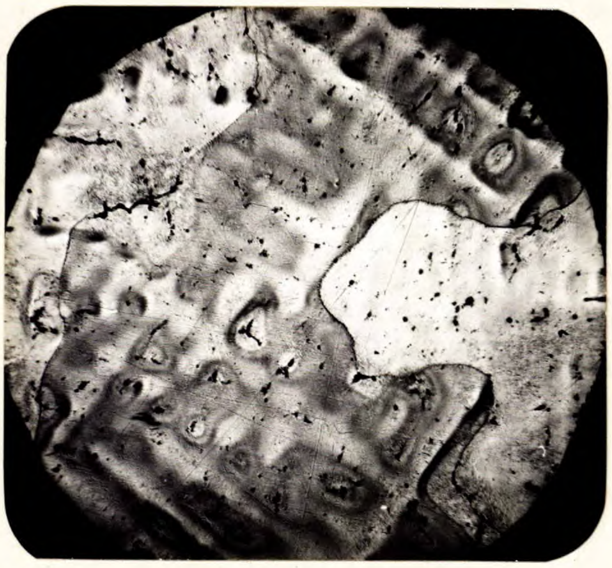
X25
RETORT NO. 3162-B-1.
2000° F. - 10 hrs., W.Q.

Figure 18.



X25
RETORT NO. 3162-B-1.
2100° F. - 20 hrs., W.Q.

Figure 19.

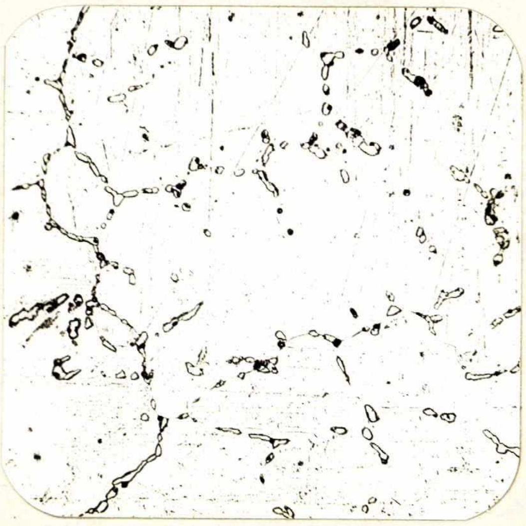


X25
RETORT NO. 3162-B-1.
2200° F. - 10 hrs., W.Q.

Figure 20.

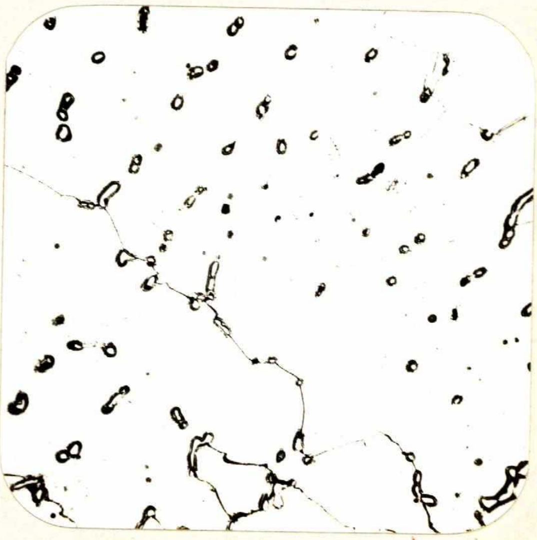
X25.
RETORT NO. 3162-B-1.
2300° F. - 10 hrs., W.Q.
Note complete absence
of segregations.
Compare with Figures 17, 18 and 19.

Figure 21.



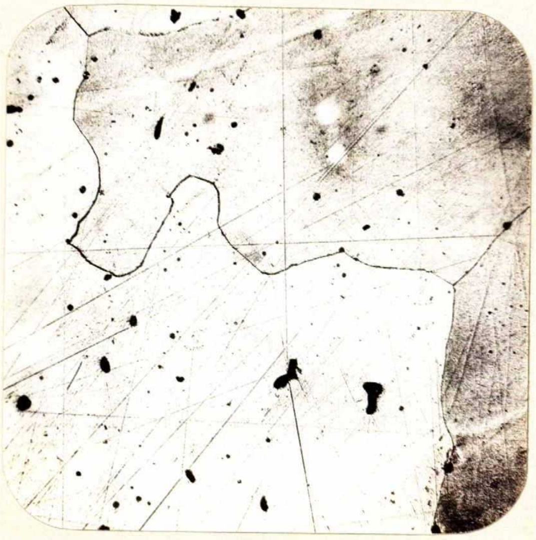
X100.
RETORT NO. 2877-B-2.
2100° F. - 20 hrs., W.Q.
Note persistence of carbides.
Compare with Figure 8.

Figure 22.



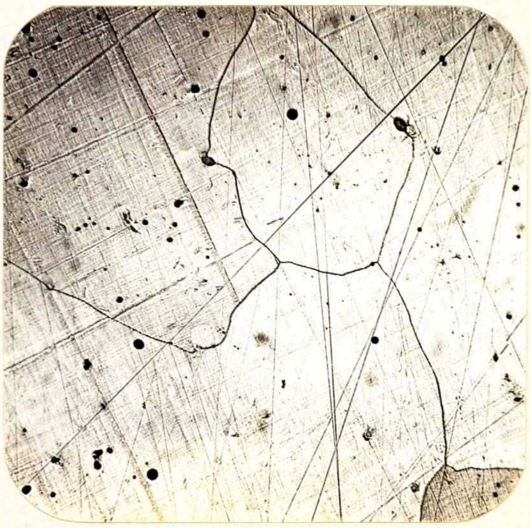
X100.
RETORT NO. 2877-B-2.
2200° F. - 10 hrs., W.Q.
Note persistence of carbides.
Compare with Figure 21.

Figure 23.



X100.
RETORT NO. 2877-B-2.
2300° F. - 10 hrs., W.Q.
Note absence of carbides.
Compare with Figures 21 and 22.

Figure 24.

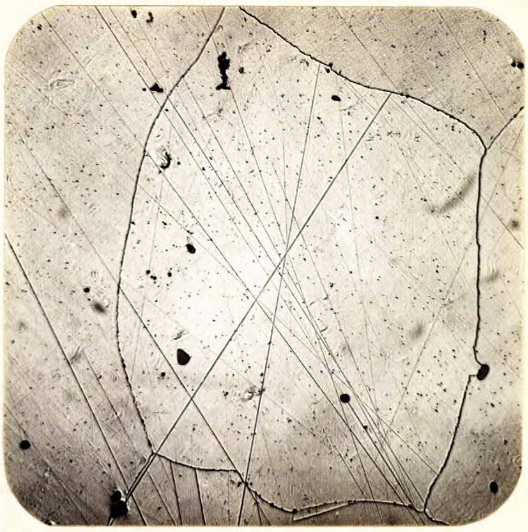


X100.
RETORT NO. 2905-B-2.
2300° F. - 10 hrs., W.Q.
Note absence of carbides.
Compare with Figure 10.

Figure 25.

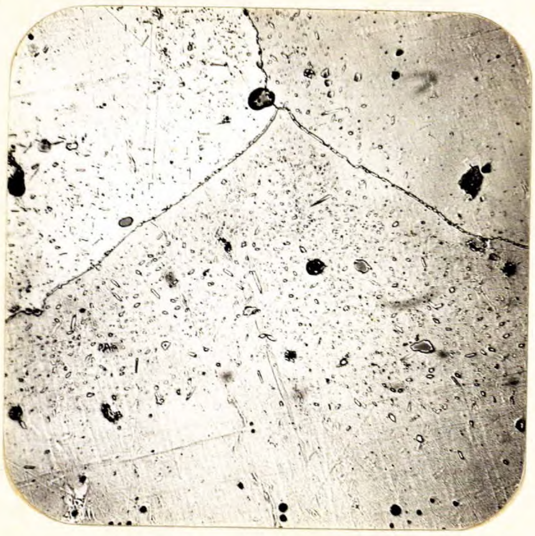
X100.
RETORT NO. 2728-B.
2300° F. - 10 hrs., W.Q.
Note absence of carbides.
Compare with Figure 6.

Figure 26.



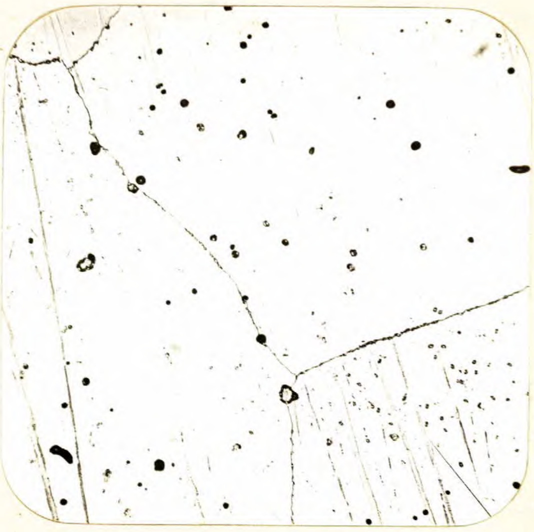
X100.
RETORT NO. 3162-B-1.
2300° F. - 10 hrs., W.Q.
2100° F. - 10 hrs., W.Q.
Note absence of carbides.
Compare with Figure 20.

Figure 27.



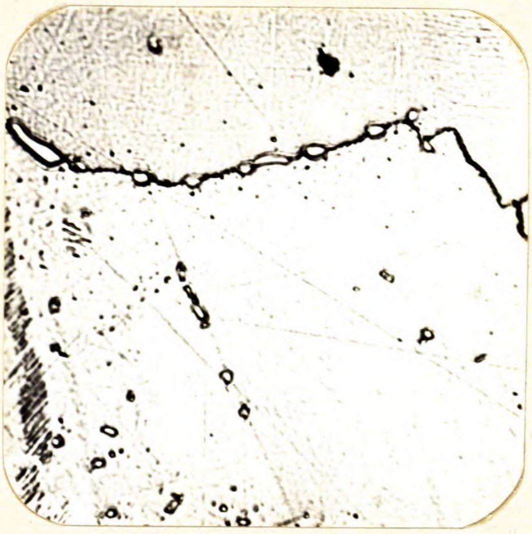
X100.
RETORT NO. 2905-B-2.
2300° F. - 10 hrs., W.Q.
2100° F. - 10 hrs., W.Q.
Note precipitation of carbides.
Compare with Figure 24.

Figure 28.



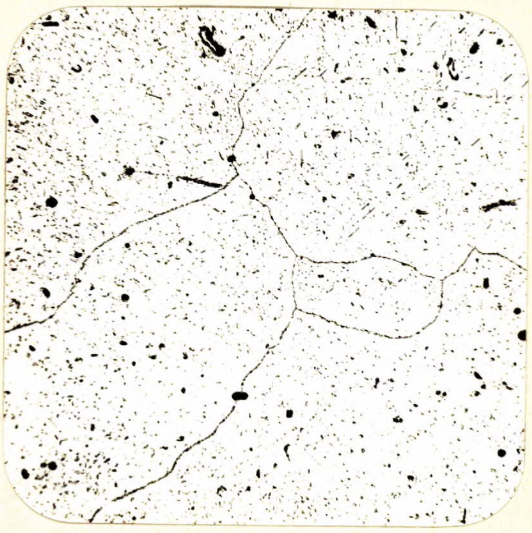
X100.
RETORT NO. 2728-B-2.
2300° F. - 10 hrs., W.Q.
2100° F. - 10 hrs., W.Q.
Note precipitation of carbides.
Compare with Figure 25.

Figure 29.



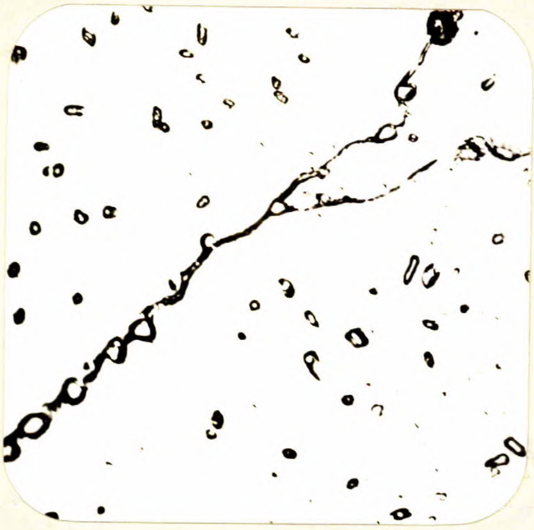
X1000.
RETORT NO. 2728-B-2.
Same as Figure 28.

Figure 30.



X100.
RETORT NO. 2877-B-2.
2300° F. - 10 hrs., W.Q.
2100° F. - 10 hrs., W.Q.
Note precipitation of carbides.
Compare with Figure 23.

Figure 31.



X1000.
RETORT NO. 2877-B-2.
Same as Figure 30.

HVK:GHB.