O T T A W A April 13th, 1944.

R E P O R T

of the

ORE DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 1627.

Examination of Section from a Stainless Steel, Centrifugally Cast Retort which Had Failed in Service.

(Copy No. 10.)

Bureau of Mines
Division of Metallic
Minerals.

Ore Dressing
and Metallurgical
Laboratories

CANADA
DEPARTMENT
OF
MINES AND RESOURCES
Mines and Geology Branch

OTTAWA April 13th, 1944.

REPORT

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Examination of Section from a Stainless Steel, Centrifugally Cast Retort which Had Failed in Service.

Origin of Request and Object of Investigation:

A small section of a retort which had failed prematurely in service at Dominion Magnesium Limited, Haley, Ontario, was received on March 21st, 1944, from the manufacturer, Shawinigan Chemicals Limited, Steel Division, Shawinigan Falls, Quebec.

In a covering letter, dated March 16th, 1944, Mr. C. K. Lockwood, of the Stainless Steels and Alloys Division, Shawinigan Chemicals Limited, Power Building, Montreal, Quebec, requested that an examination be made to determine the cause of failure.

Origin of Material:

This centrifugally cast retort (No. C-846, Heat No. 3660-B-1), which failed after 37 days of service, was of 25/20 composition (25 per cent chromium, 20 per cent nickel). Other retorts of the same composition have given

(Origin of Material, cont'd) -

good service, 120 days or more to date, in the furnace. In his request letter, Mr. Lockwood stated that the failure was not due to scaling but was apparently due to cracks developing in both the top and bottom sections of the retort. The cracking developed directly over the central support.

Macroscopic Examination:

The section submitted measured approximately 9½ x 7½ x 1 inch. Figures 1 and 2 are photographs of the section in the "as received" condition. The cracking is plainly shown in these photographs. Figure 3 is a photograph of a deep-etched section (etched in 2 per cent picric, 10 per cent HCl in ethyl alcohol for ½ hour). Note, in this photograph, the increased grain size at the top of the section, indicating extent of decarburized zone.

Chemical Analysis:

The chemical analysis determined by these Laboratories is shown below. The analysis made by Shawinigan Chemicals Ltd., is also given, for comparison.

Analysis		Bureau of Mines	Shawinigan Chemicals
		- Per cent -	
Carbon	-	0.25	0.24
Manganese	-	0.80	0.88
Silicon	-	0.43	0.48
Nickel	-	21.97	20.33
Chromium	-	24.20	24.06
Molybdenum	-	0.03	-
Vanadium	-	0.06	-
Copper	-	0.18	Nil*

* Qualitative test.

Preparation of Microspecimens, and Heat Treatment:

Three samples, A, B and C, cut from the section of retort submitted, were given the following preparation:

Sample A was prepared for examination in the "as received"

(Preparation of Microspecimens, and Heat Treatment, cont'd) -

condition.

Sample B was heat-treated as follows:

(1) Heated to 1160° C. (2120° F.),
(2) Held at this temperature for 5 hours,
(3) Quenched in water.

Sample C was heat-treated as follows:

(1) Heated to 1200° C. (2192° F.),
(2) Held at this temperature for 5 hours,
(3) Quenched in water.

All three samples were polished by hand and then etched with the following reagent:

1 gram picric acid,
5 grams hydrochloric acid,
100 cc. ethyl alcohol.

Sample B was heat-treated at 1160° C. (2120° F.) because this was the working temperature of the retort, and the structure at the working temperature was desired for comparison with the "as received" structure.

Sample C was heat-treated at 1200° C. because the heat treatment given Sample B produced a carbide precipitation. It was necessary to determine whether or not carbide precipitation could be produced by heating to the maximum known operating temperature of the retort. This heat treatment would indicate whether or not the retort had been overheated.

Microscopic Examination:

Sample A (As Received) -

This sample was hand polished, etched, and examined microscopically. The structure revealed was wholly austenitic with some free massive carbides mainly on the grain boundaries. There was a tendency towards segregation of the carbides. Figure 4, at a magnification of 100 diameters, shows this section under bright field (normal) illumination. Figure 5, at a magnification of 200 diameters, is a photomicrograph taken

(Microscopic Examination, cont'd) -

on the same sample (but a different field) under dark field illumination.

Sample B (Heat Treated at 1160° C.) -

A section of the retort, after being heat-treated as described above, was hand polished, etched, and examined microscopically. The structure was wholly austenitic, with free massive carbides chiefly on the grain boundaries, and also showing a marked precipitation of carbides within the grains. There was a tendency towards segregation of the carbides in this section (as in the "as received" section). Figure 6, at a magnification of 100 diameters, shows a section of this sample under bright field illumination. Figure 7, at a magnification of 200 diameters, shows a section of this sample under dark field illumination. There was considerable carbide precipitation in this sample, very clearly shown in Figure 7.

Sample C (Heat Treated at 1200° C.) -

A section of the retort, after heat treating as described above, was hand polished, etched, and examined microscopically. The structure revealed was wholly austenitic with free massive carbides on the grain boundaries and no precipitation of carbides within the grains. Figure 8, at a magnification of 100 diameters, shows a section of this sample under bright field illumination, while Figure 9, at a magnification of 200 diameters, shows a section of the sample under dark field illumination. The similarity of the "as received" structure with the structure after heating at 1200° C. is evident from a comparison of Figures 5 and 9.

Discussion of Results:

The chemical analysis agrees fairly closely with the specifications of 20 per cent nickel, 25 per cent chromium. The

(Discussion of Results, cont'd) -

analysis also agrees with that made by Shawinigan Chemicals Limited.

The microscopic examination showed a completely austenitic structure containing some carbides in all three sections examined (as received, heat treated at 1160° C., and heat treated at 1200° C.). This is the normal structure for steel of 20 per cent nickel, 25 per cent chromium composition.

It has been shown (Investigation No. 1560, dated January 11th, 1944) that when a piece of steel of similar structure but having a different Ni-Cr ratio is heated to a sufficiently high temperature and quenched from that temperature, most of the carbides will go into and remain in solution in the austenite. A subsequent heating to a lower temperature will result in a precipitation of these carbides. This is illustrated in Figures 17 and 18 of Report of Investigation No. 1560, which are reproduced as Figures 10 and 11 of this report. Obviously the size and degree of dispersion of these carbides will depend on the length of time the piece is held at the precipitation temperature.

Specimen B showed a precipitation of carbides when heated to 1160° C. This shows that the austenite had more carbides in solution than it could hold at this temperature.

For the same reason the absence of precipitation after heating Specimen C to 1200° C. indicates that the solubility of the austenite for carbides at that temperature was high enough to retain the dissolved carbides in solution. This means that these carbides were taken into solution at some temperature between 1160° and 1200° C. Had this retort been heated to a temperature above 1200° C. in service, there would also have been a precipitation of carbides in

(Discussion of Results, cont'd) -

Specimen C. Since it is known that, if blow-up be also included, the present practice at the Dominion Magnesium Limited plant at Haley is to operate these retorts in this temperature range, it is evident that this retort was not heated outside the normal range.

CONCLUSIONS:

In view of the fact that the data for this report were obtained from an examination of a very small section of the retort, it is possible that there are some factors involved in the operation of this retort, unknown to the writer, that could explain the failure.

The chemical analysis and microscopic examination showed that the steel used in this retort was satisfactory both as to composition and to structure.

The retort had not been heated outside the normal range.

The indications are that the retort failed at a temperature in the neighborhood of 1200° C. It is known that the retorts are blown up at a temperature of 1190° C., but examination of the section of retort submitted indicated that the retort had not failed during a blow-up; the cracking and buckling took place in the wrong direction, that is, inwards. It would seem likely that the failure might be due to operating the retort for too long a period before blowing it up.

The failure might also have some connection with the experiments being conducted in furnace operation at temperatures in the neighbourhood of 1200° C.

There would appear to be no metallurgical reason for the premature failure of this retort.

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Figure 1.

SECTION OF RETORT, SHOWING FAILURE.
"AS RECEIVED" CONDITION.
Cracks are on inside wall of retort.
(Approximately 1/3 size).

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Figure 2.

SECTION OF RETORT, SHOWING FAILURE.
"AS RECEIVED" CONDITION.
(Approximately ½ size).

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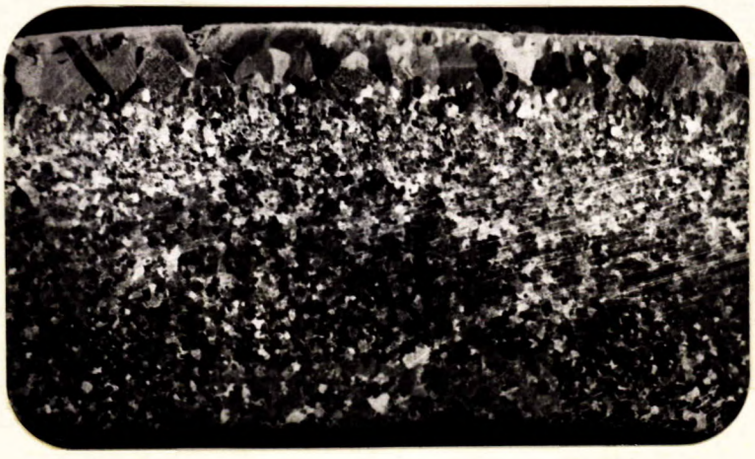
Figure 3.



DEEP-ETCHED SECTION OF RETORT.
Note increased grain size at top of section.
(Approximately twice actual size).

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Figure 4.

X100, etched.

SAMPLE A (AS RECEIVED).

Wholly austenitic structure with some massive carbides at grain boundaries.

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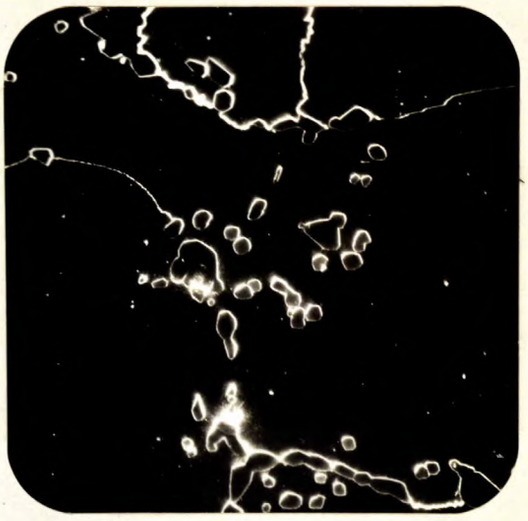
Figure 5.



X200, etched.

SAMPLE A (AS RECEIVED).

Dark field illumination. Note absence of precipitation of carbides.

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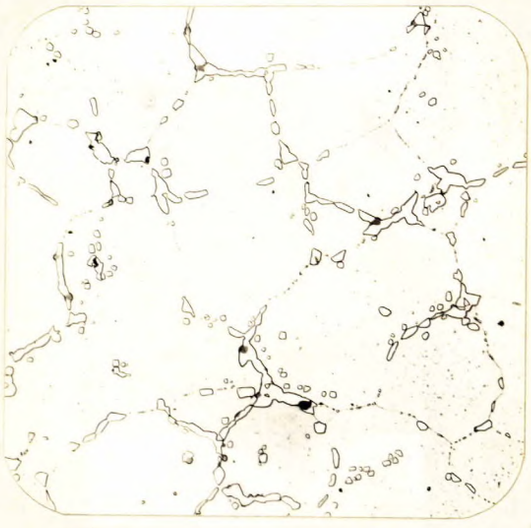
Figure 6.



X100, etched.

SAMPLE B
(HEAT TREATED AT 1160° C.)

Wholly austenitic structure, massive carbides at grain boundaries, and carbide precipitation within grains.

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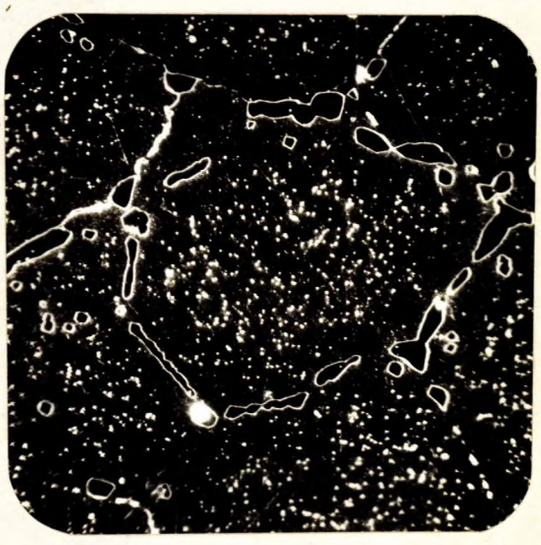
Figure 7.



X200, etched.

SAMPLE B
(HEAT TREATED AT 1160° C.)

Dark field illumination. Note heavy carbide precipitation.

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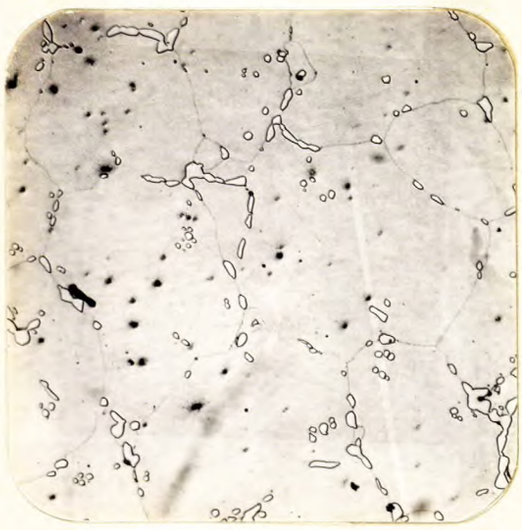
Figure 8.



X100, etched.

SAMPLE C
(HEAT TREATED AT 1200° C.).

Wholly austenitic structure with massive carbides at grain boundaries.

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Figure 9.

X200, etched.

SAMPLE C
(HEAT TREATED AT 1200° C.).

Dark field illumination. Note absence of carbide precipitation.

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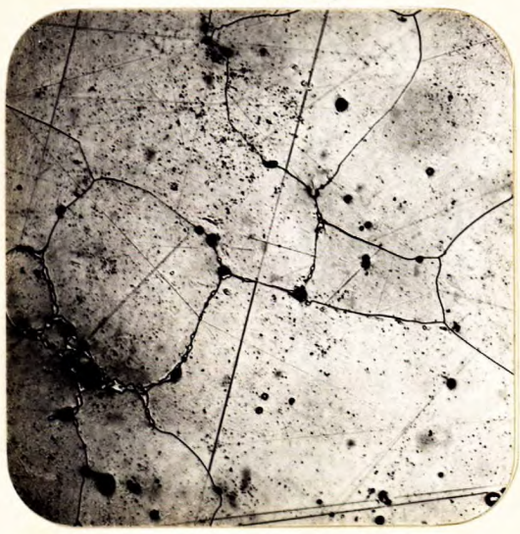
Figure 10.



PHOTOMICROGRAPH, X100, OF SPECIMEN TAKEN FROM RETORT NO. 2787-B-2.

Typical structure when held at 2300° F. (1260° C.) for 10 hours and water-quenched. Note that most carbides have disappeared.

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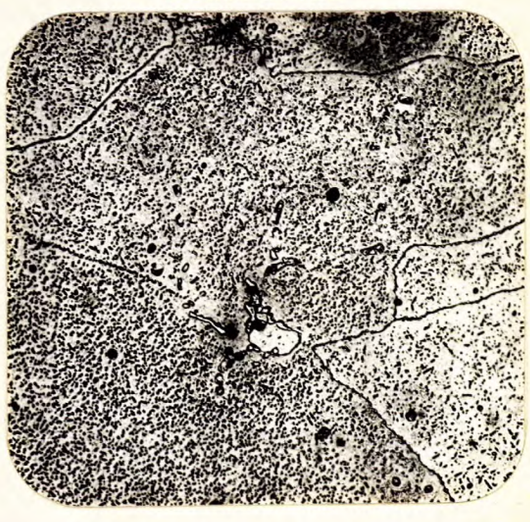
Figure 11.



PHOTOMICROGRAPH, X100, OF SPECIMEN TAKEN FROM RETORT NO. 2787-B-2.

Typical structure when reheated to 2130° F. (1165° C.) and water-quenched after water quenching from 2300° F. (1260° C.) Note precipitation of carbides.

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