File.

FILE COPY

O T T A W A July 25th, 1944.

R E P O R T

of the

ORE DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 1690.

Examination of Corroded Cap from High Alloy Steel Retort Used in Magnesium Production.

(Copy No. 10.)

Bureau of Mines
Division of Metallic
Minerals
—
Physical Metallurgy
Research Laboratories

CANADA
DEPARTMENT
OF
MINES AND RESOURCES
Mines and Geology Branch

O T T A W A July 25th, 1944.

R E P O R T

of the

ORE DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 1690.

Examination of Corroded Cap from High Alloy Steel Retort Used in Magnesium Production.

Origin of Material:

On June 6th, 1944, the Stainless Steel and Alloys Division of Shawinigan Chemicals Limited, Shawinigan Falls, Quebec, submitted a part of a retort (C-904) which had been returned from Dominion Magnesium Limited. The retort had failed after only 28 days of service, the failure being the complete corrosion of the weld joining the cap to the retort barrel. This failure is characteristic of an increasing number, and is therefore the cause for some concern.

The part submitted consisted of the complete cap, weld, and approximately one inch of the barrel of the retort.

Object of Investigation:

To determine the cause of the weld corrosion and to suggest means of its elimination.

Procedure:

1. The corrosion product of the weld was subjected to chemical analysis, with the following results:

		Per Cent
F_2	-	0.09
Chromium	-	11.93
Nickel	-	25.64
Copper	-	0.03
Iron	-	32.63

If the above elements are combined with oxygen to give the following oxides, Cr_2O_3, Fe_3O_4 and Ni_3O_4, and the percentage of metallic elements calculated, it can be shown that on addition 98.05 per cent of the weight of the scale is accounted for. It is, of course, axiomatic that the corrosion product will contain metallic elements in the same proportion as was present in the original weld metal.* A second calculation indicates that the weld metal contained approximately 35 per cent nickel and approximately 15 per cent chromium.

2. The cap material was subjected to chemical analysis, with the results listed below:

		Per Cent
Carbon	-	0.14
Phosphorus	-	0.018
Sulphur	-	0.013
Manganese	-	0.35
Silicon	-	0.42
Chromium	-	17.89
Nickel	-	38.33
Molybdenum	-	Trace.
Copper	-	0.07
Titanium	-	Trace.
Columbium-	-	None.

3. Macro sections were machined from two areas. Figure 1 shows a section of the retort barrel and the almost complete corrosion of the weld. Figure 2 shows another section of the retort barrel, weld and cap material. In this case approximately one-half of the weld depth has corroded away. In both cases the large grains show the extent of decarburization.

(Continued on next page)

* See O.D.M.L. Report of Investigation No. 1524, November 1st, 1943.

(Procedure, cont'd) -

4. Both of the above macro sections were repolished, etched, and subjected to a microscopic examination. All corroded surfaces showed even attack, with no preferential oxidation along grain boundaries.

5. A section of the cap material approximately 6 inches square was ground smooth and a 1-inch-diameter hole was drilled in each corner to a depth of ½ inch. Flux was removed from Arcos Chromend 15/35 (Arcos Corp.) and Arcaloy Type 330 (Alloy Rods Co.) electrodes. This flux was weighed out in 1-gram lots. Two holes of the test plate were charged with 1 gram of Arcos flux and the other two with Arcaloy flux. The peripheries of the holes were then stamped respectively with the numbers 1 and 2. Figure 3 shows the test plate at this stage of the procedure.

The test plate was then heated to 2150° F. in an electric furnace and held at that temperature for 48 hours. At the end of that period the plate was removed from the furnace and photographed within one minute. This is necessary to photograph the oxides in situ, since they fracture off as the plate is cooled. Figure 4 shows the appearance of the plate while the temperature was in excess of 2000° F.

The plate was allowed to cool slowly to room temperature and then was wire-brushed to remove loosely adhering scale. Figure 5 shows the appearance of the plate after this treatment.

6. Samples of these fluxes were subjected to chemical analysis. The following table lists the results secured:

(Continued on next page)

(Procedure, cont'd) -

		Arcos Flux	Arcaloy Flux
		- Per Cent -	
F_2	-	17.40	8.06
SiO_2	-	17.38	9.68
CO_2	-	15.92	10.56
Manganese	-	2.52	5.35
$CaCO_3$	-	19.75	12.63
Total calcium as CaO	-	44.50	22.88
MgO	-	1.30	Trace.
MoO_3	-	None.	5.45
Na_2O	-	3.72	2.83
K_2O	-	None.	None.
TiO_2	-	None.	31.92
Nickel	-	Trace.	Trace.

Discussion:

The analysis of the corrosion product of the weld indicates the use of a 35 Ni/15 Cr type of electrode. This is good welding practice in that there is a close match with the composition of the retort material. This provides uniform physical properties and corrosion resistance around the weld area. The copper content may be a residual or the result of dilution from the cap material during welding.

The analysis of the cap material reveals nothing unusual. The composition is of the 35 Ni/15 Cr type, the copper content being too low to have any deleterious effect on corrosion resistance. It is well to notice, in passing, that this alloy has not been stabilized with either titanium or columbium.

The macro and micro examinations reveal no evidence of possible causes of corrosion. It is interesting to note (see Figure 2) that the recommended joint design (a U joint) has not been used but, rather, a simple V joint with an included angle of 50 degrees and a zero root gap. This type of joint is too narrow and difficulties might be encountered in slag removal. The microscopic examination reveals no evidence of preferential oxidation along grain boundaries that

(Discussion, cont'd) -

is characteristic of stress corrosion. The attack seems to be characterized by general dissolution over the entire affected areas of the type generally seen in chemical attack. The rather severe decarburization and accompanying grain growth have no known effect on resistance to corrosion.

The results of the corrosion tests at high temperature are significant and in agreement with others* investigating this problem. It is apparent that the test conditions duplicate fairly closely the operating conditions of the magnesium retorts. It would appear that at 2150° F. both fluxes have a liquid phase, differing only in amount. The Arcos electrode flux has by far the greater amount of this liquid phase at temperature, and this has extremely high wetting power and fluidity as evidenced by the climbing from the drilled hole and spreading over a wide surface. The photograph (Figure 4) taken while the test plate was about 2000° F. shows the extremely heavy ring of corrosion product around the holes containing the Arcos flux. It is noteworthy that the holes containing the Arcaloy flux have no such severe attack. It has been shown that the corrosive damage caused by the liquid phase is out of all proportion to the amount in action. The mechanism is, apparently, that the liquid phase acts as a solvent on the protective oxide coating, thereby continuously exposing fresh surfaces.

Of the large number of possible components of electrode coatings the low-melting-point fluorides are the main constituents of the damaging liquid phase which, together with silicates, can cause very severe corrosion. As shown above, the Arcos flux contains by far the greater amounts of both silicates and fluorides. In view of this analysis and the results of the high temperature corrosion test, there is every

---

* Geiger - Research Development Laboratories, International Nickel Co. Report of Jan. 15, 1943.

(Discussion, cont'd) -

reason to believe that the fluxes have been responsible for the corrosion of the material submitted for examination.

Removal of welding fluxes on the completion of a pass is a far more difficult procedure than is generally recognized. Electrode manufacturers, in recognition of these difficulties, frequently employ a rutile (TiO_2) type of electrode coating. This produces a slag which is very friable and frequently will spontaneously crack away cleanly from the weld on cooling. The other main type of electrode coating is known as the "lime" type and contains large quantities of calcium carbonate. These "lime" coatings produce a friable slag which, however, tends to powder under the points of chipping tools and leave a fine deposit between the ripples of beads. To most welders this adherent powder is of no importance, and it is difficult to bring them to a full realization of the damage it may cause. In the case of slag entrapped within a weld, there is no opportunity for the liquid phase to spread out over a large surface. The damaging corrosion is confined to relatively small areas in the first phase and spreads only as metal is dissolved. This would account for complete corrosion of a weld in one area and only partial corrosion in another area of the same weld.

From the analysis of the two fluxes it will be noted that the Arcos flux is of the "lime" type, whereas the Arcaloy flux is of the rutile type.

CONCLUSIONS:

1. The corroded weld was made using a 35 Ni/15 Cr type of electrode.

2. The retort cap is of an unstabilized 35 Ni/15 Cr

(Conclusions, cont'd) -

composition.

3. The weld joint was a simple V with a 50 degree included angle and zero root gap, and not the recommended U joint.

4. The corrosive action of the Arcos flux is far greater than that of the Arcaloy flux. This is due to the high fluorine and silicate contents of the former. It would appear that the weld corrosion has been due to welding flux trapped in the weld.

5. The Arcaloy electrode coating is of the rutile type and that of the Arcos of the "lime" type.

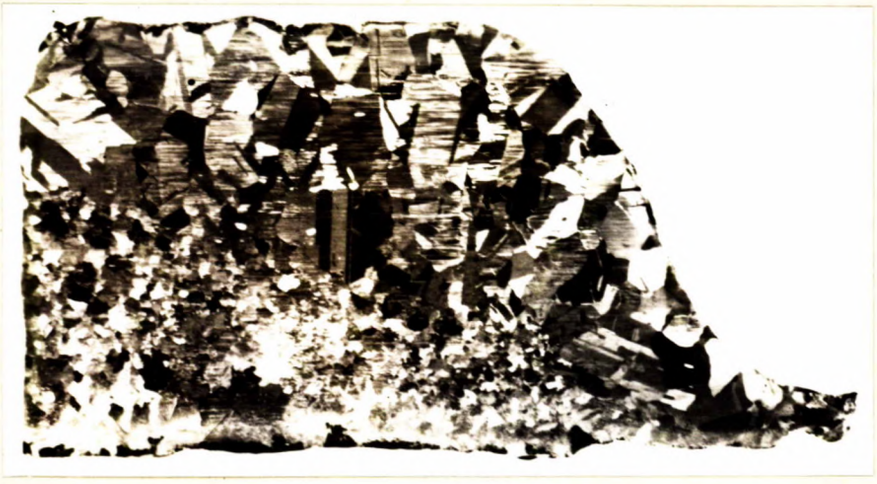
Recommendation:

Welding of retorts should be done with an electrode with a rutile-type coating which is low in fluorine and silicate contents.

oooooooooo
ooooo
o

HJN:LB.

Figure 1.

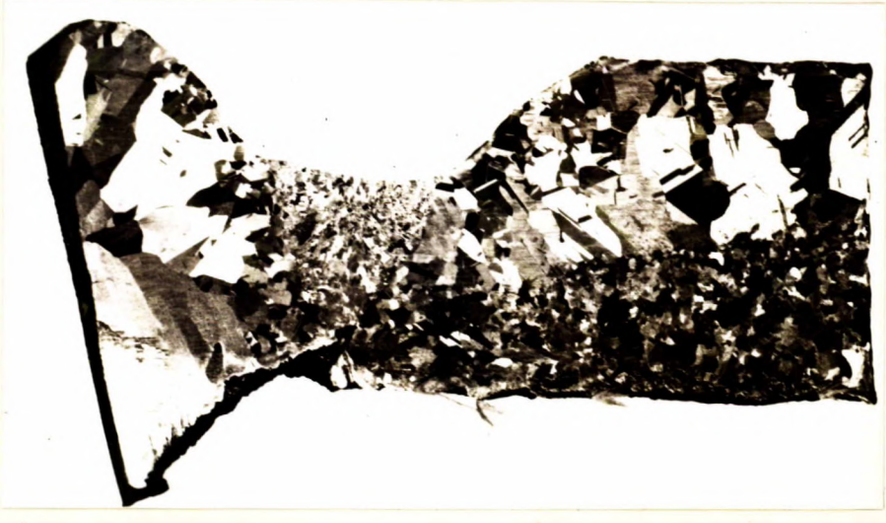


X2¼, etched in aqua regia.

MACRO SECTION OF RETORT AND WELD.

Note large grain size resulting from decarburization.

Figure 2.



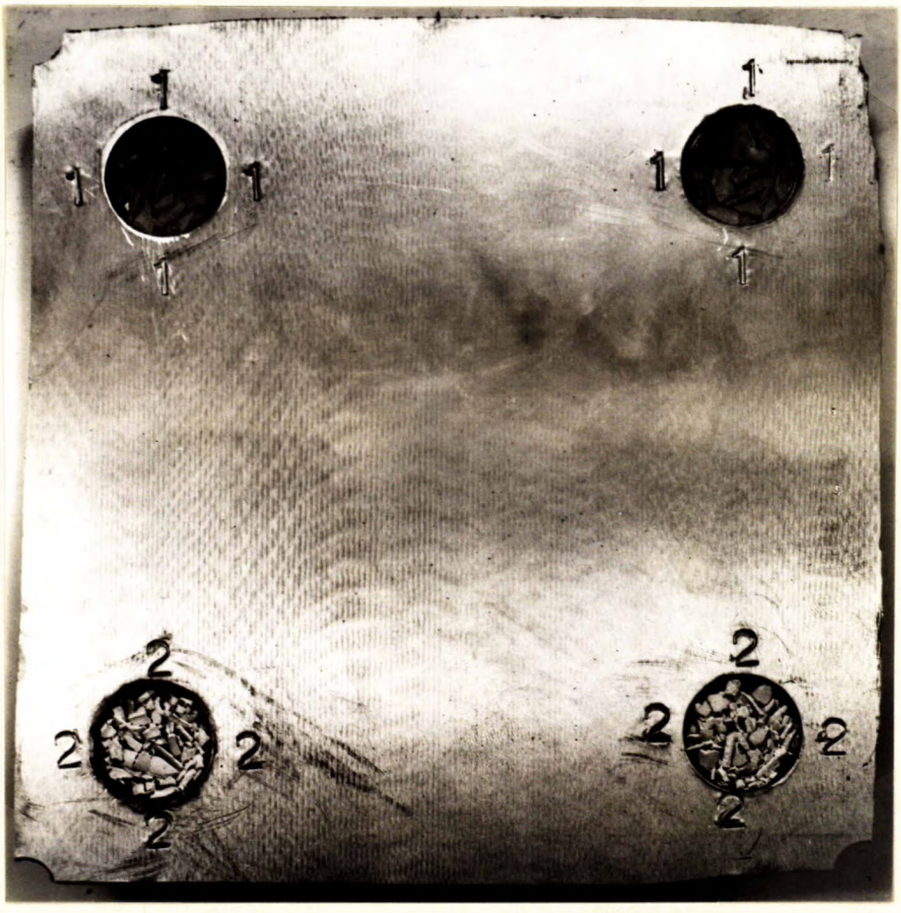
X2, etched in aqua regia.

MACRO SECTION OF RETORT, WELD AND CAP MATERIAL.

Note the large grain size resulting from decarburization. Note also weld outline revealing a V type of joint with zero root gap.

Figure 3.

use as H.J.H.'s copy

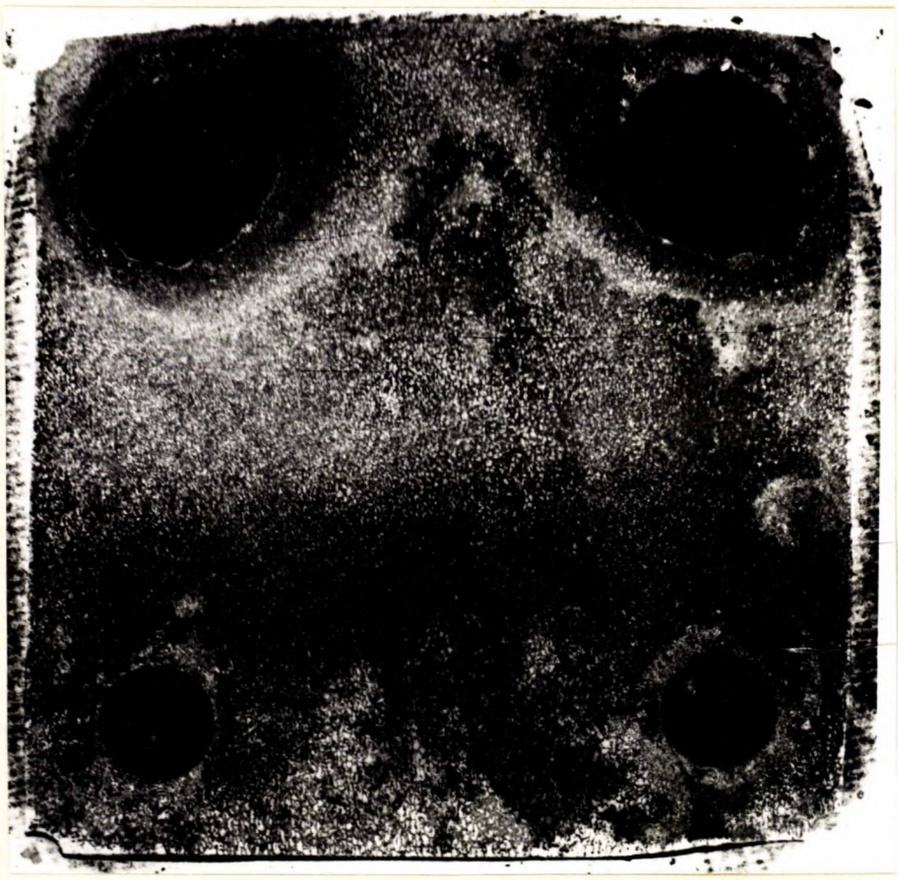


HIGH TEMPERATURE CORROSION TEST PLATE.

Section of Retort Cap, Ground Smooth.

The holes are 1 inch in diameter and ¼ inch deep.
Holes numbered 1 contain 1 gram of Arcos flux;
those numbered 2 contain 1 gram of Arcaloy flux.

Figure 4.



HIGH TEMPERATURE CORROSION TEST PLATE
AT TEMPERATURE OVER 2000° F.

Holes number 1 are at top; holes numbered 2 are at bottom.

Note heavy ring of corrosion product around holes containing Arcos flux. Note also wide ring around each top hole, revealing extent of spread of liquid phase. Bottom holes (Arcaloy flux) show slight evidence of a liquid phase also.

—

Figure 5.

HIGH TEMPERATURE CORROSION
TEST PLATE WHEN COLD.

Plate Wire-Brushed When Cold.

Note severe surface pitting around top holes. Note also heavy scaling of top holes.

HJN:IB.