UNCLASSIFIED.

Copy No. 1 of 7 Copies.
D.M. 682/54.
RB 37/78

DEPARTMENT OF MINES

SOUTH AUSTRALIA.

PRELIMINARY INVESTIGATIONS INTO CORROSION OF STAINLESS STEELS DURING LEACHING OF URANIUM CONCENTRATES FROM RADIUM HILL. S.A.

SUMMARY:

Corrosion tests were carried out on several types of stainless steel to determine their suitability for use in construction of components for digesters for the leaching of Radium Hill concentrates. The testing was carried out under actual leaching conditions with varied initial acid concentrations. Concentrates were leached both as received and after washing to remove chloride which was expected to affect corrosion rates materially.

It was found that in every instance corrosion was greater in the presence of larger amounts of chloride, and that when lower initial acid concentrations were used corrosion was very severe when concentrate leached contained only 0.2% chloride

It is recommended that stainless steel should not be used for the construction of digester components. If no alternative to stainless steel can be found then it will be necessary to wash all concentrates to remove chloride in order to protect the stainless steel from corrosion.

H E St Pierre.
Experimental Officer.

HEStP:AK.
12/4/54.

A. INTRODUCTION:

During the operation of the pilot plant for treatment of uranium concentrate from Radium Hill, it was noticed that corrosion of the stainless steel parts of the digesters was accelerated when leaching methods were changed to the use of more dilute sulphuric acid. To enable the design staff to decide whether stainless steel could be used with safety in the construction of agitators for the digesters in the production plant at Port Pirie, and, if so, what type of stainless steel would be most suitable, corrosion testing of various stainless steels was undertaken.

It was proposed to determine relative corrosion rates under the two sets of leaching conditions that had been recommended as alternatives for the extraction of uranium, and also to gauge the effect, upon corrosion rates, of the small percentage of chloride present in the concentrate as produced at Radium Hill. This chloride is introduced into the concentrate as sodium chloride in the water used in the metallurgical processing.

Subsequently, corrosion rates were also determined using a modification of the second set of leaching conditions.

B. EXPERIMENTAL PROCEDURE:

A glass lined kettle fitted with a steam jacket and a reflux condenser was used in which to carry out leaching tests embodying the several variations of charge. The corrosion test pieces were in each case tied to the stirrer which rotates at 100 R.P.M.

A number of test pieces were cut from sheet of four different types of stainless steel and a fresh piece was used for each variation of corrosive conditions. Cutting was done by hacksaw to avoid excessive heating of the metal. Surfaces were made as clean and smooth as possible. Test pieces were weighed before and after each leach in which they were immersed

and corrosion rates were estimated according to losses in weight. Visual observations of corrosion effects were also made.

Leaching conditions were:-

(1) Boiling for six hours with sulphuric acid of an initial concentration of 53% (w/w) at 52% solids (w/w) in the leach.

(2) Boiling for 12 hours with sulphuric acid of an initial concentration of 37% (w/w) at 50% solids (w/w) in the leach.

(3) Modification of (2) above. Namely, boiling for 12 hours with sulphuric acid of an initial concentration of 46% (w/w) at 55.5% solids (w/w) in the leach. This uses the same weight of sulphuric acid as in (2). (750 lbs/short ton concentrate.)

Using conditions (1) and (2), leaches were carried out on concentrates containing the normal amount of chloride as produced, and also concentrates which had been water washed to remove chloride. In the "washed concentrate" leaches distilled water was used to make up the charge, while in the "normal chloride concentrate" leaches, tap water was used which contained approximately 0.1 grams/litre chloride.

In one leach, employing the more dilute acid, the charge was made up by using "backwash Liquor" from the pilot plant, which is a dilute liquor returned to circuit, instead of water. The object was to have a solution containing at least 1 gram/litre of ferric iron in the leach before the addition of the acid. The presence of ferric iron in solution is claimed to inhibit the corrosion of stainless steel by sulphuric acid.

A further leach was carried out using live steam for heating with no reflux condenser, as this might allow the escape of chloride as hydrochloric acid gas and thus alter corrosion rates.

The leach with sulphuric acid of an initial concentration of 46% (w/w) at 55.5% solids (w/w) had the object of obtaining leach liquor with characteristics similar to the leach with 37%

acid (w/w) at 50% solids (w/w), and at the same time approach the corrosion characteristics of the 53% acid (w/w) leach at 52% solids (w/w). This leach was on concentrates containing normal amounts of chloride.

C. RESULTS:

The results of the tests are shown in Table No. 1.

Corrosion rates have been calculated, according to the weight losses, as average depth of penetration in inches per year. In cases where corrosion was severe it always took the form of pitting, so that, in use, the life of a vessel made of such stainless steel plate would be far shorter than that indicated by the average corrosion rate. It would probably be fair to assume that corrosion takes this form even when the rate of attack is very slow, so that it would never be wise to attempt to calculate the useful life of any steel, under the corrosive circumstances tested, according to the average corrosion rates.

It should also be borne in mind that a very limited number of leaches have been carried out for the determination of corrosion rates under each set of conditions, therefore the figures reported represent the initial corrosion rates only.

The four stainless steels were labelled C.P.P., M.O., S.F. 25 and S.T.B. respectively, and had the following compositions:-

1. C.P.P. - 19.6% Cr, 9.89% Ni, 1.4% Mo, 0.08% Ti. (Analysis)
2. M.O. - 18% Cr, 8% Ni, Mo stabilised. (Specification)
3. S.F.25 - 19% Cr, 9% Ni, 1.7% Mo, Ti stabilised. (Specification)
4. S.T.B. - 18% Cr, 8% Ni, Ti stabilised. (Specification)

D. CONCLUSIONS:

The following important conclusions can be drawn from the results obtained.

1. Chloride has a very severe accelerating effect upon corrosion of stainless steels under conditions used for leaching

of Radium Hill uranium concentrate. This effect is very much greater when sulphuric acid of lower initial concentration is used for leaching.

2. All the steels tested can be regarded as "fully resistant" to corrosion (rates less than 0.042 inches penetration per year) when concentrate has been water washed to remove chloride before leaching. However, the following reservations are made regarding the above statement:-

(a) the extent to which removal of chloride must be taken to prevent severe corrosion is not known.

(b) the effect of welding upon corrosion rates of stainless steels is not known.

(c) corrosion rates reported represent initial attack only, and rates could increase subsequently.

3. When the chloride content of the concentrate is reduced by washing, all of the four steels tested are equally resistant to corrosion.

4. When corrosion is severe no one steel shows most resistance, but the steel labelled S.T.B. containing 18% Cr, 8% Ni and Ti stabilised is always the least resistant.

5. Ferric iron in solution appears not to inhibit corrosion of stainless steel by sulphuric acid when a small amount of chloride is present.

6. It appears that corrosion could be reduced somewhat, though insufficiently, when chloride is present in normal amounts, and sulphuric acid is used for leaching at the rate of 750 lbs. per short ton of concentrate, by:-

(a) using live steam for heating, with no reflux.

(b) using a higher % solids in the leach to give a higher initial acid concentration.

E. RECOMMENDATIONS:

It would be extremely hazardous to instal agitators, constructed of stainless steel, in the digesters for the leaching of Radium Hill concentrates in the treatment plant at

Port Pirie.

If there is no alternative to the use of stainless steel for construction of agitators, then, irrespective of the initial acid concentration used in leaching, corrosion will be severe unless concentrates are washed. If the lower acid concentration leach is used the corrosion would be quite intolerable unless concentrates are washed to remove chloride.

If washing of concentrates is undertaken, the allowable degree of contamination by chloride in the concentrates (which has not yet been established) must never be exceeded or the expensive stainless steel equipment could be ruined.

Other precautions to see that chloride is not admitted to the digesters would be necessary.

T A B L E N O. I.

LEACHING CONDITIONS FOR CORROSION TESTS.	Chloride Content of Radium Hill Concentrate Leached	Number of Leaches Test Pieces Subjected To	Label Denoting Type of Stainless Steel	Average Corrosion Rate in Inches Penetration per Year	COMMENTS
Leach at 52% solids (w/w) with initial sulphuric acid concentration of 53% (w/w). Boiling for 6 hours. B.P. temperature 120°C - 110°C	As received		C.P.P.	0.043	No visible signs of corrosion
	0.12		M.O.	0.224	No visible signs of corrosion on plain surfaces, but slight etching of edges.
	and	2			
	0.20		S.F. 25	0.044	No visible signs of corrosion.
	% Cl		S.T.B.	1.993	Marked etching of surfaces
Leach at 50% solids (w/w) with initial sulphuric acid concentration of 37% (w/w). Boiling for 12 hours. B.P. temperature 113°C - 105°C	As received		C.P.P.	1.85	Corrosion mostly around edges, and only several small pittings on plain surfaces
	0.23				
	% Cl	1	M.O.	2.46	Corrosion mostly around edges, but also much pitting of plain surfaces
			S.F. 25	1.83	Corrosion mostly around edges, but also several large pittings on plain surfaces
			S.T.B.	6.26	Whole test piece honeycombed with deep pittings.
Leach at 52% solids (w/w) with initial sulphuric acid concentration of 53% (w/w). Boiling for 6 hours. B.P. temperature 120°C - 110°C	Water Washed		C.P.P.	0.0066	No visible signs of corrosion
	0.02		M.O.	0.0037	No visible signs of corrosion
	and	2	S.F. 25	0.0027	No visible signs of corrosion
	0.05 % Cl		S.T.B.	0.0042	No visible signs of corrosion
Leach at 50% solids (w/w) with initial sulphuric acid concentration of 37% (w/w). Boiling for 12 hours. B.P. temperature 113°C - 105°C.	Water Washed		C.P.P.	0.0010	No visible signs of corrosion
			M.O.	0.0029	No visible signs of corrosion
	0.06 % Cl	1	S.F. 25	0.0017	No visible signs of corrosion
			S.T.B.	0.0033	No visible signs of corrosion
Leach at 50% solids (w/w) with initial sulphuric acid concentration of 37% (w/w). Boiling for 12 hours. B.P. temperature 113°C - 105°C. "Backwash" used instead of water in charge so as to have at least 1 gram/litre of Fe^{+++} in solution before acid addition	As received		C.P.P.	2.83	Corrosion mostly around edges, but also one plain surface badly pitted.
	% Cl not determined	1	M.O.	3.18	Corrosion mostly around edges, but also much pitting of plain surfaces.
			S.F. 25	2.50	Corrosion mostly around edges, but also one plain surface badly pitted.
			S.T.B.	10.43	Whole test piece honeycombed with deep pittings, and little of original surface remaining.
Leach at 50% solids (w/w) with initial sulphuric acid concentration of 37% (w/w). Boiling for 12 hours. B.P. temperature 113°C - 105°C. Heated by live steam and using no reflux condenser	As received		C.P.P.	0.35	Some corrosion around edges, but plain surfaces essentially free of pitting
	% Cl not determined	1	M.O.	1.88	Corrosion mostly around edges, with a few small pittings on plain surfaces.
			S.F. 25	1.29	Corrosion mostly around edges, with several large pittings on plain surfaces
			S.T.B.	3.88	Many deep pittings on all surfaces

LEACHING CONDITIONS FOR CORROSION TESTS.	Chloride Content of Radium Hill Concentrate Leached	Number of Leaches Test Pieces Subjected to	Label Denoting Type of Stainless Steel	Average Corrosion Rate in Inches Penetration per Year	COMMENTS
Leach at 55.5% solids (w/w) with initial sulphuric acid concentration of 46% (w/w). Boiling for 12 hours. B.P. temperature 120°C - 108°C.	As received		C.P.P.	0.52	Some corrosion visible around edges
	0.15 % Cl	1	M.O.	0.82	Corrosion mostly around edges, but several small pittings on plain surfaces
			S.F. 25	1.16	Pittings most numerous around edges, but also some deep pittings on plain surfaces.
			S.T.B.	4.30	Deep pittings frequent on all surfaces.