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# Construction and Operation of a Laboaratory Size Thum Cell

Raymond Dugdale

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Construction and Operation of a Laboratory Size Thum Cell

by

Raymond Dugdale

A Thesis

Submitted to the Department of Metallurgy

in Partial Fulfillment of

the Requirements for the Degree of

Bachelor of Science in Metallurgical Engineering

Montana School of Mines

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## Introduction

Electrolytic silver refining was not perfected until the end of the nineteenth century. During the process of development, two systems of silver refining have come into prominence: the Moebius and the Thum types. The Moebius cell is characterized by the use of vertical anodes surrounded by canvas bags that act as a diaphragm to catch the gold slimes and also by the use of silver starting sheets as cathodes. The Thum cell uses a horizontal sheet of carbon or stainless steel at the bottom of the cell for the cathode and the anodes are suspended horizontally above the cathode in a canvas lined basket. Usually, both systems are used together because the Moebius process leaves some crude silver scrap and the Thum process is especially adapted to use this scrap material.

Many modifications have been made on both processes, an example of which is the use of a conveyor belt cathode in the Thum cell to regulate the height to which the silver crystals would grow and to remove them from the main cell in a certain time interval. This is done to save the amount of labor that was needed to keep the crystals knocked down and removed as in the original process. Other changes have been made, but the United States mints and most other refineries in the United States have kept the original systems with little modification. The usual installation may be as large as 180 cells, but a ratio is maintained of about a dozen Moebius cells to one or two Thum cells. The cells that are used in the San Francisco mint measure 19 by 39 inches by 18 inches deep. These cells are made of brown earthenware and the baskets for the Thum cells are made entirely of maple wood which is put together without



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the use of nails or metals of any kind.

### Theory

The Thum cell is operated using from 24 to 100 amps per sq. ft. fo cathode surface depending on the gold content of the anode. A high gold content warrants the use of lower current densities and vice versa. At som mints, no diaphram is used on those cells that are parting high gold anodes. The gold is strong enough to support itself after parting and there is no contamination of the cathode deposit if a low current density is maintained.

A simple ion bath of silver nitrate is used for the parting and the silver content varies from fifteen to sixty grams per liter of solution according to the individual plant practices. Most plants also use from fifteen to sixty grams of copper per liter to increase the conductance of the parting bath. This increased conductance gives a product of stubbier crystals.

Because there is no need for a bright or adherent deposit, no attention is paid to the deposit except for the crystal growth. The crystals that grow in a pure silver nitrate bath are very long and needlelike and grow at a fast rate. These long crystals soon short the cell and excesseve labor would be required for efficient operation. Thus the copper is added to help start the cell operating at near nomal conditions, but there is copper in the anode as impurity that enters the solution as parting proceeds and the copper content of the bath increases to such a high amount that some of the electrolyte must be removed and replaced with pure silver nitrate solution.

The impurities in the anode are usually copper, lead, arsenic, antimony,

1. Edward B. Durham, Trans. A.I.M.E., Bulletin 58, p.814.



the platinum group metals, and other minute quantities of the other elements. All of the platinum group metals are insoluble in the bath and are deposited with the gold on the diaphragm and thus removed and recovered in another precious metals process. The lead, copper and other impurities pass into the electrolyte and are subsequently removed with the silver before the solution is discarded.

### Problem

The problem was to produce a Thum cell for laboratory use, using an anode 1 by 2 by one eighth inches and also using voltages and currents the same as used in practice. It was decided that an anode containing 5% copper, 2% lead, 0.27% gold with the remaining amount silver would be representative. A current of 50 amps per sq. ft. and 3.5 volts was also decided upon. Since this decision, many references that were consulted quoted figures that varied from 1.5 to 5 volts and a variance in current from 12 to 100 amps per sq. ft. Mostly this variance was determined by the gold or impurity content of the anodes.

The following things had to be accomplished to solve the problem: (1) to produce or find a suitable receptacle to use as a cell, (2) to produce an anode to use in the cell, and (3) to put these two together to fit the established ends.

An old battery case, which was 5 inches deep and small enough to fit the need, was obtained for the cell. The carbon cathode was fit to the bottom of the cell and also a mold was made out of carbon to cast the anode. The canvas was procured from the assay office to be used for the diaphragm. After a suit-

2. W.A. Koehler, Electrochemistry Vol. II, Second ed., (1944) p.178.

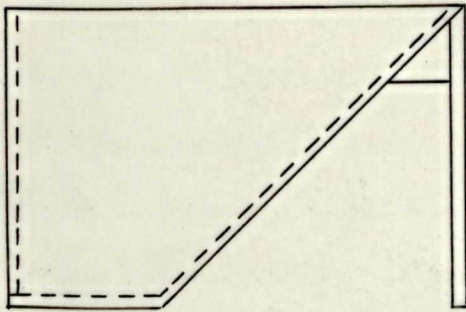


able anode was obtained, the cell was set up using a pure silver nitrate bath in order to determine the spacing between the electrodes. This turned out to be  $2\frac{1}{2}$  inches at the required current and voltage. It was decided that the carbon was too porous because the silver was imbedded in the carbon so that it could not be removed without ruining the cathode. A stainless steel cathode was substituted for the carbon and proved very satisfactory in use.

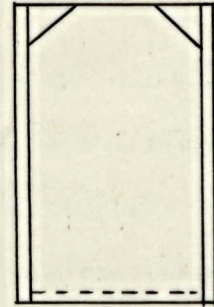
During this time, a source of lucite plastic material was discovered and the decision was made to use this material to construct the cell (see Fig. 1, p.5) and pieces were cut from the plastic sheets. These pieces were welded together using a solution of lucite powder in glacial acetic acid with a little acetone added. This method proved very satisfactory, and a cell was made without any difficulty. The next problem was to make a basket out of lucite. This proved to be just as easy a task as the cell construction was because of the amazing workability of the material and the ease with which the plastic is welded together with the lucite solution.

A standard voltmeter was connected across the electrodes and a standard ammeter was attached in the negative lead. A ten ohm slide wire resistor was used in conjunction with a 6 volt copper oxide rectifier to obtain the required voltage. The rectifier had a continuous current rating of 2 amps and was the type that plugged directly into the laboratory 110 VAC line and stepped the voltage down to 6 volts across the rectifiers. These articles were connected with the cell to make a permanent unit for the next work (see Fig. 2, p.6). The anode was suspended in the basket on the canvas bag and the stainless steel cathode was modified to fit the new cell. The cathode surface now measured 3.75 sq. inches.

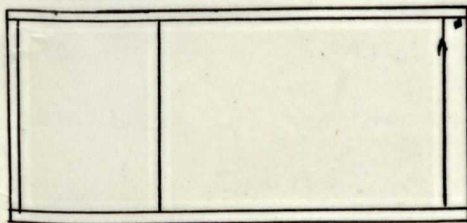




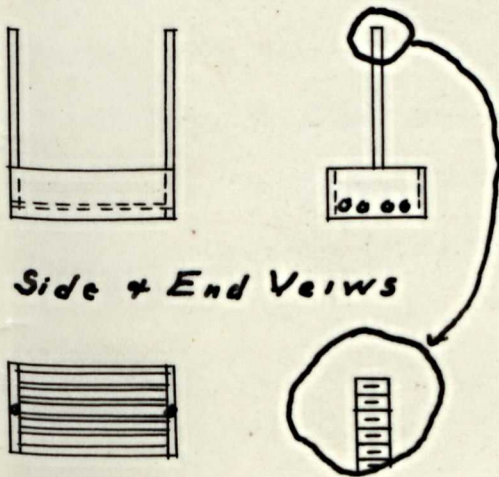
Side View



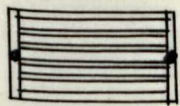
End View



Top View



Side & End Views



Top View

### Materials

#### Cell

Sides  $\frac{1}{4} \times 3\frac{1}{2} \times 7\frac{1}{4}$

End  $\frac{1}{4} \times 3\frac{1}{4} \times 2\frac{1}{2}$

Incline  $\frac{1}{4} \times 6\frac{3}{8} \times 2\frac{1}{2}$

Bottom  $\frac{1}{4} \times 2\frac{1}{2} \times 2\frac{1}{8}$

#### Basket

Sides  $\frac{1}{8} \times \frac{5}{8} \times 2\frac{1}{4}$

Ends  $\frac{1}{8} \times \frac{5}{8} \times 1\frac{1}{2}$

Rods  $\frac{1}{8}$  round  $\times 2\frac{1}{2}$

Supports  $\frac{1}{8}$  round  $\times 3$

Bolts -  $\frac{1}{4}$  sheet

Fig. 1.- Cell



### Cell Operation

The pure silver nitrate solution containing 60 gr. of silver per liter was added to the cell so that it just covered the anode when it was suspended  $2\frac{1}{2}$  inches from the cathode and the current was applied. The current was adjusted to approximately 0.7 amps for the first two runs. This was a mistake made by the author in determining the current density on the basis of anode surface and not cathode surface. The current was adjusted to the calculated 1.3 amps for the last run as a correction. Thus the current density for the first two operations was only 27 amps per sq. ft. of cathode surface and 50 amps per sq. ft. for the last run.

The cell was operated for 40 minutes during the first test. The crystals started to grow uniformly over the cathode and after about one eighth inch of crystal growth, most of them stopped growing entirely and only a few scattered crystals continued. These active crystals were long and needlelike and proceeded to grow toward the anode at a fast rate. After these crystals would get to a certain height, they would break off and fall to the bottom of the cell. Before long, these crystals had enough support from the fallen parts so that they had to be broken off or they would soon short the cell. A glass probe was used to do this.

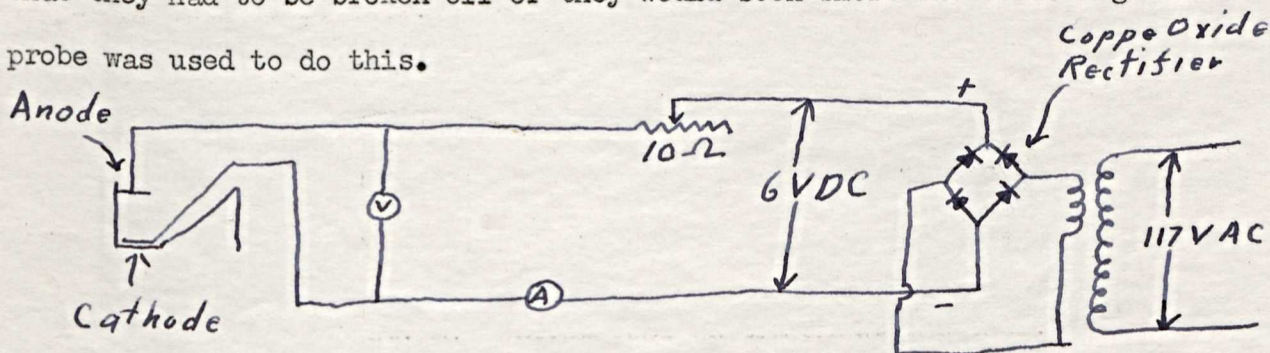


Fig. 1. Circuit used in operating the cell.



A microscopic examination was made of the cathode deposit to attempt to find the reason why the silver had deposited in such a few trees and why it didn't deposit uniformly over the cathode as far as the building of the trees went. The product looked burnt under the microscope where the trees that had started had not continued to grow. An examination of the literature showed that this condition may be caused either by the presence of basic salts or that an unstable oxide is formed that stops deposition. The unstable oxide could be controlled by the addition of small amounts of alcohol to the bath.<sup>3</sup> The presence of basic salts would be a remote possibility but it was more logical to try the alcohol first because of the care with which the electrolyte was made. This assumption proved to be true because the oxide was not formed in the next run. The first run produced 1.61 grams of silver whereas the anode decreased 1.53 grams in weight. Part of this loss in weight was caught in the diaphragm as slime, so less than 95% of the silver came from the anode.

The same electrolyte was used during the second run. Thus the copper content was building up in the solution and during this run, the electrodes had to be moved to a distance of two and three fourths inches. A few drops of alcohol were added to the electrolyte and as was stated above, the burnt condition was corrected by this action. The crystals were much more platelike in appearance and stubbier, which proved that as the copper content of the bath increased, the conductance increased, and this caused the crystals to plate out heavier and stubbier. The second operation produced 2.653 grams of sil-

3. Eric K. Rideal, Industrial Electrometallurgy, (1919) p.80.



ver after an hour while the anode decreased 2.6164 grams part of which was slime in the diaphragm. Thus, less than 98.5% of the silver came from the anode. It can be seen that the electrolyte was being depleted of silver and building up in acid content as the time of operation was increased.

The third test was run for 3 hours and 45 minutes using the electrolyte from the previous test. As was stated above, 1.3 amps were used this time. During this test, the crystals were even more platelike and very little difference in crystal growth was noted during the first part of the run. The crystals were very bright and even textured. As the crystals grew, the anode was moved upward to compensate for their growth. An electrode spacing of one inch was found to be the right value for this amperage and after the electrolyte had reached a constant temperature of 52 degrees centigrade. Near the end of the test, fairly active convection currents were at work and the spots where these currents seemed to concentrate started to build up toward the anode. This test produced 18.662 grams of silver and the anode decreased by 18.535 grams which showed less than 99% of the silver came from the anode.

Data

Test	Anode Weight Before	After	Loss	Silver Deposit
I	48.6756 gr.	47.1364 gr.	1.5392 gr.	1.61 gr.
II	47.1364 gr.	44.52 gr.	2.6164 gr.	2.653 gr.
III	44.52 gr.	25.985 gr.	18.535 gr	18.662 gr.

The cell reached a constant temperature of 52 degrees centigrade after 1½ hours at 50 amps per sq. ft.

Alcohol prevented the oxide coating of the crystals.



### Results

As stated above, The gradually increasing copper content made the silver crystals grow heavier and stubbier. This would aid in parting where the electrodes were separated a fixed distance. With the cell as it is though, the electrodes can be separated as the crystals grow and thus eliminate the need for removing the silver from the cell until the end of the test. If it is noted that the copper content is too high, a simple addition of more silver nitrate would solve that problem without having to shut the cell down,

The cell itself withstood the temperature of the electrolyte and no other affects wer noted. The plastic was supposed to withstand acids and bases with the exception of acetic acid. The bonding material that was used was not attacked in the use of the cell and stayed impervious to the electrolyte.

Without accurate instruments to measure the current of the cell, it would be difficult to determine the efficiency of the cell so no attempt has been made to do so. The literature stated that the efficiency should be 100%, but with the formation of nitric acid in the cell, there is no way of attaining such a high efficiency. Therefore, an accurate test should be made of the current efficiency but the time didn't permit.

The overall operation of the cell was very satisfactory, and the project of construction of the cell was aided immensely by having such a wonderful material as lucite to use.

Without the addition of a few drops of alcohol every so often, the deposit probably wuld not have turned out as good as it did during the last run. Therefore, whenever the cell is used, alcohol should be added to insure the optimum operation.

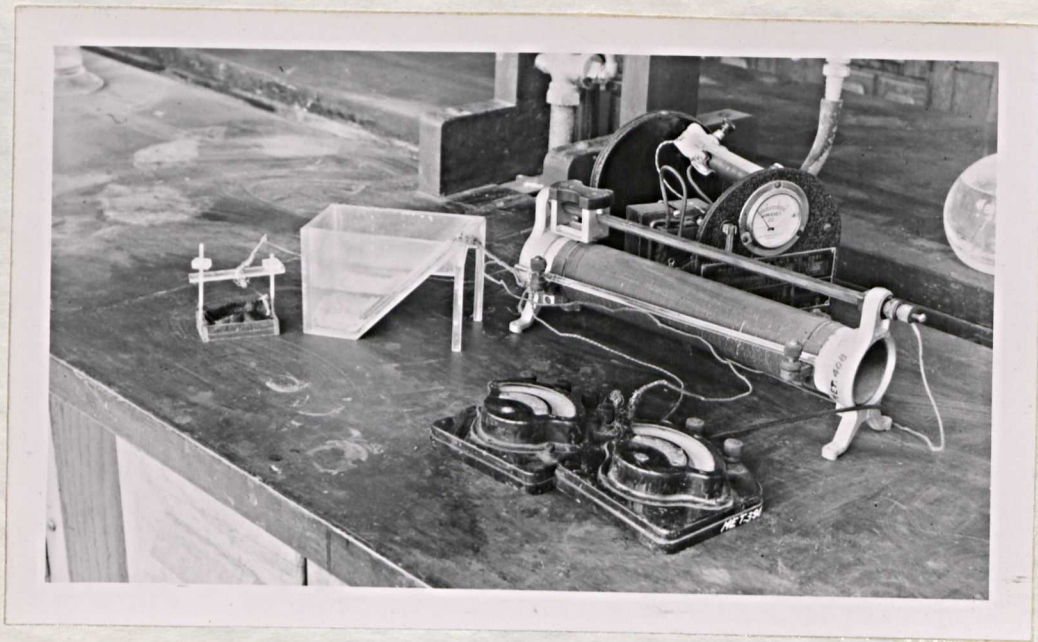


### Conclusions

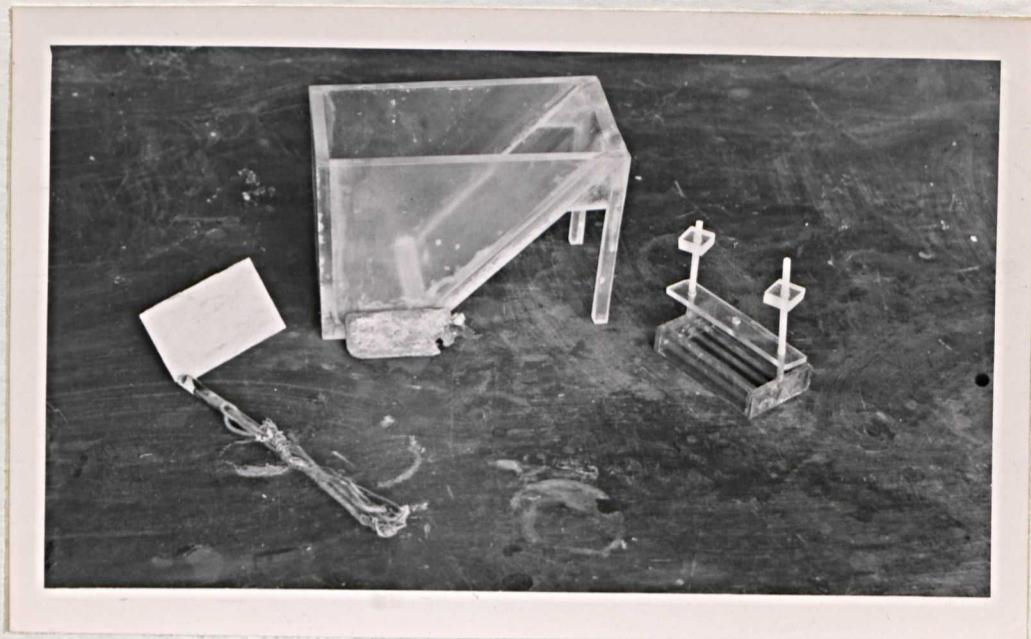
1. The cell should operate without much difficulty if the electrolyte contains some copper to begin with, but not enough to hamper the normal operation of the cell. This addition of copper would stop the fast growth of the crystals at first and thus cause less need for attention to the cell. Alcohol should be added a few drops at a time as it evaporates or if uneven crystal growth is obtained. If the crystals should grow too fast, they should be knocked down with the rake that is made for this purpose.

2. The cell will withstand the temperature that is caused by the 50 amp per sq. ft. current density, but the softening temperature of the plastic is 66 degrees centigrade and also the plastic goes through thermal deformation between 50 and 85 degrees centigrade. Therefore, it would not be advisable to operate at a much higher temperature,





Permanent set up for operating the Thum cell.



The component parts of the laboratory Thum cell.



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