AUG 3 1964

MASTER

RI bureau of mines
report of investigations 6489

ELECTROREFINING BERYLLIUM

Operation of a Prototype Cell

By M. M. Wong and J. E. Klosterman

PATENT CLEARANCE OBTAINED. RELEASE TO
THE PUBLIC IS APPROVED. PROCEDURES
ARE ON FILE IN THE RECEIVING SECTION.

UNITED STATES DEPARTMENT OF THE INTERIOR

BUREAU OF MINES

1964

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

ELECTROREFINING BERYLLIUM

Operation of a Prototype Cell

By M. M. Wong and J. E. Klosterman

Facsimile Price $ 1.60
Microfilm Price $ .80

Available from the
Office of Technical Services
Department of Commerce
Washington 25, D. C.

* * * * * * * * * * * report of investigations 6489

MARCH 3, 1849

UNITED STATES DEPARTMENT OF THE INTERIOR
Stewart L. Udall, Secretary

BUREAU OF MINES
Marling J. Ankeny, Director

The work upon which this report is based was done under a cooperative agreement between the Bureau of Mines, U.S. Department of the Interior, the U.S. Atomic Energy Commission, and the University of California Lawrence Radiation Laboratory.

This publication has been cataloged as follows:

Wong, Morton Min

Electrorefining beryllium, operation of a prototype cell, by M. M. Wong and J. E. Klosterman. [Washington] U.S. Dept. of the Interior, Bureau of Mines [1964]

17 p. illus., tables. (U. S. Bureau of Mines. Report of investigations 6489)

Based on work done in cooperation with the U. S. Atomic Energy Commission, and the University of California, Lawrence Radiation Laboratory.

1. Beryllium–Electrometallurgy. I. Klosterman, J. E., jt. auth. II. Title. III. Title: Prototype cell. (Series)

TN23.U7 no. 6489 622.06173

U. S. Dept. of the Int. Library

CONTENTS

ILLUSTRATIONS

TABLES

ELECTROREFINING BERYLLIUM

Operation of a Prototype Cell

by

M. M. Wong[1] and J. E. Klosterman[2]

ABSTRACT

Scrap beryllium chips (nominally 96 to 97 percent Be) were electrorefined in a prototype cell with 408 lb of electrolyte composed of 52.4 mole-percent LiCl, 36.3 mole-percent KCl, and 11.3 mole-percent $BeCl_2$. The cell temperature was 500° C. In a typical 10-3/4-hour deposition cycle, 0.415 pound of metal was produced; the metal contained impurities in parts per million as follows: C, 700; O, 610; Al, 34; Ca, 32; Cu, 20; Fe, 25; Mg, 9; and Mn, 23.

Operation of the prototype cell for 2 years demonstrated that adapting the electrorefining process to an enlarged scale of operation presented few difficulties in mechanical functions, maintenance of optimum operating conditions, and control of product purity.

After the spent anode feed was leached in a 5-percent nitric acid solution and the fine-sized particles were removed, it was recycled in some tests. The metal produced was somewhat higher in impurities than metal from the fresh scrap chips.

INTRODUCTION

Fused-salt electrorefining had been applied to beryllium in laboratory-scale cells by the Bureau of Mines.[3] The technique was successful as a method

---

[1] Supervisory research chemical engineer, Boulder City Metallurgy Research Laboratory, Bureau of Mines, Boulder City, Nev.

[2] Former Bureau of Mines supervisory research chemical engineer, Boulder City Metallurgy Research Laboratory, Boulder City, Nev.; now with Gilfillan Bros., Inc., Los Angeles, Calif.

[3] Wong, M. M., R. E. Campbell, and D. H. Baker, Jr. Electrorefining Beryllium. Studies of Operating Variables. BuMines Rept. of Inv. 5959, 1962, 14 pp.
Wong, M. M., F. R. Cattoir, and D. H. Baker, Jr. Electrorefining Beryllium. Preliminary Studies. BuMines Rept. of Inv. 5581, 1960, 9 pp.

Work on manuscript completed November 1963.

of preparing high-purity beryllium or as a process for recovering beryllium from scrap metal.

The operating variables, such as electrolyte composition, temperature, cell voltage, cathode current density, and cell geometry, had been investigated previously.[4] The work described in this report consists of an electrorefining operation in a prototype cell to study the scaled-up operation and to produce high-purity metal for research uses in the University of California Lawrence Radiation Laboratory.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the assistance and advice given by E. R. Adkins, R. F. Bunshah, W. M. Miller, and V. N. Karpenko of the University of California Lawrence Radiation Laboratory.

SELECTION OF OPERATING CONDITIONS AND CELL DESIGN[5]

On the basis of information obtained from previous work,[6] the following operating conditions were chosen for the prototype cell: (1) an electrolyte of 52.4 mole-percent lithium chloride (LiCl), 36.3 mole-percent potassium chloride (KCl), and 11.3 mole-percent beryllium chloride ($BeCl_2$); (2) a cell temperature of 500° C, and (3) a protective atmosphere of inert gas. Under these operating conditions, beryllium was deposited as crystals.

The requirements that governed cell design were prevention of contamination of the product, effective utilization of electrode area, and easy removal of electrodes from the cell during operation.

Product metal could be collected either by withdrawal on a suspended cathode or by harvest from the electrolyte chamber surfaces which served as the cathode. Both methods would require removal of the product through an inert-gas lock to prevent its oxidation while cooling and to prevent contamination of electrolyte by contact with the air. The suspended-cathode method was selected because harvest of the product from the electrolyte chamber offered greater possibility for metal contamination by sludge that would accumulate in the bottom of the cell and because a simpler air lock system was possible.

Laboratory-scale research had shown that beryllium had a pronounced preference to deposit on the cathode area nearest to the anode feed. For efficient utilization of cathode area and symmetry of deposit configuration, an anode-cathode relationship with maximum surface areas parallel to each other was desirable.

A means for replacement of the anode feed after an appropriate degree of depletion was essential to maintain product quality and to minimize the

---

[4] Works cited in footnote 3.

[5] MacMullin, Robert Burns. The Problem of Scale-Up in Electrolytic Processes. Electrochem. Tech., v. 1, No. 1-2, January-February 1963, pp. 5-17.

[6] Works cited in footnote 3.

accumulation of sludge in the cell system. Spent anode feed acquired surface coatings that diminished the activity of the remaining beryllium, thus resulting in the transfer of impurities to the cathode during electrolysis.

Two cell types appeared to be suitable: (1) concentric cylindrical electrodes and (2) parallel flat electrodes. The first cell type offered a symmetrical and very compact anode-cathode configuration, but it made periodic withdrawals of the peripheral electrode for material removal cumbersome. The second cell type did not utilize the cell volume as efficiently as the first cell type, but it was simple to operate and flexible in permitting expansion of the number of electrode pairs in further scaling-up of the operation.

In selecting the cell size for a desired production capacity, two characteristics of a beryllium deposit were taken into consideration: (1) the low bulk density of the crystal growth, which required ample space, and (2) the weak adherence of the crystal to the cathode, which limited the size of the deposit that could be recovered by withdrawal without too much falling off. These two factors were more important than the IR drop of the electrolyte in deciding anode-cathode spacing.

Requirements for materials of construction were not overly critical. Mild steel was satisfactory for the exterior of the cell, and nickel was excellent for the parts that were in contact with the molten salt. Copper could be used as the electrode leads, but it would cause a slight contamination of the product. Therefore, nickel was used instead, although it was not as good an electric conductor as copper.

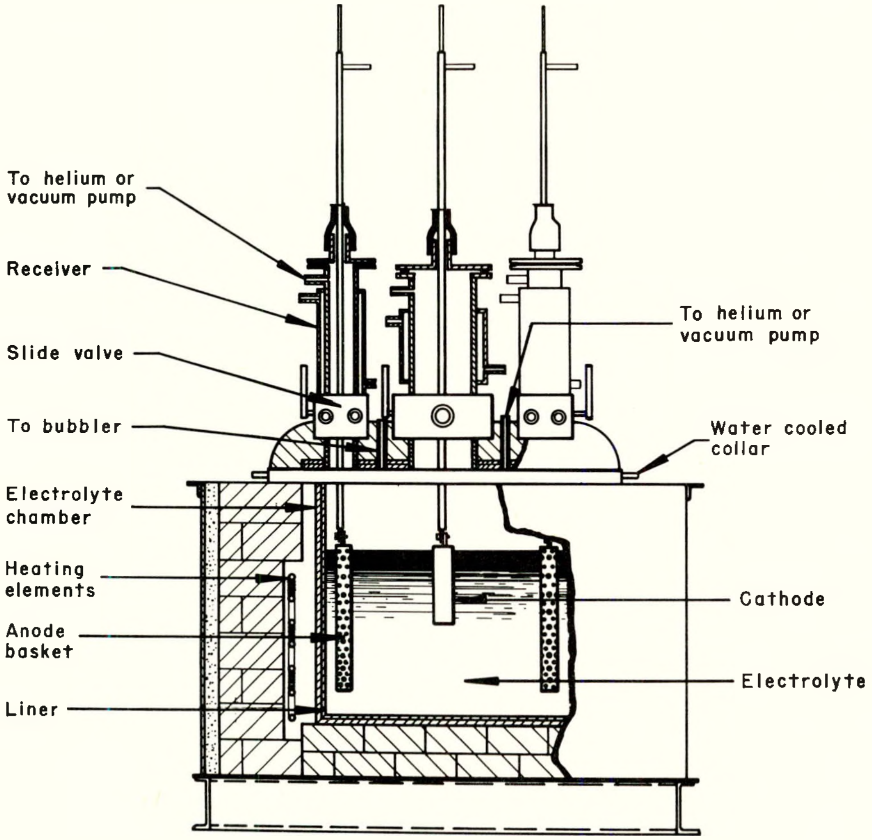

FIGURE 1. - Schematic Drawing of the Prototype Cell.

EQUIPMENT

A schematic drawing of the prototype cell is shown in figure 1. The cell consisted of an electrolyte chamber and a cover assembly. The rectangular electrolyte chamber was constructed of 1/2-inch-thick mild steel with the following outside dimensions: 31 inches wide, 15 inches long, and 27 inches high. A 1-1/2-inch-wide flange was welded to the top edges. An 11-gage nickel crucible (liner) was fitted inside the chamber, and a metallized coating was applied to the outside of the chamber.

The cover assembly was fabricated from mild steel and contained three water-cooled receivers. The middle receiver, with inside dimensions of 8 inches wide, 12 inches long, and 16 inches high (later it was extended 4-1/2 inches), was used to accommodate the cathode; two receivers with inside dimensions of 3 inches wide, 12 inches long, and 7-1/2 inches high were used to accommodate the anodes, one on each side of the cathode. A water-cooled slide valve was installed between each receiver and the electrolyte chamber.

The electrolyte chamber and the cover assembly were welded together before installation in the furnace, as shown in figure 2. After the cell was installed in the furnace, a water-cooled collar was positioned below the flange of the electrolyte chamber and on the top of the furnace. An insulating material was applied over the top of the electrolyte chamber, and a safety hood was mounted over the receivers.

The electrode leads were made of 3/4-inch-OD nickel tubes. They were internally water cooled and were inserted into the cell through rubber sleeves and fittings in the covers of the receivers. The center-to-center spacing between the anode and cathode was 12-1/2 inches.

Figure 3 shows the cathode and the anode basket. The cathode was made of 16-gage nickel with an oblong base of 5-1/2 inches in one axis and 1-1/2 inches in the other. It was 7-1/2 inches high with an 11-gage nickel handle welded to the top. The bottom of the cathode was open.

The anode baskets were shaped like the cathode and were 11 inches in the major axis, 2 inches in the minor axis, and 15 inches high. They were made of perforated 16-gage nickel with perforations 1/8 inch in diameter and 25 holes per square inch. The top was equipped with an 11-gage nickel handle, and the bottom was closed.

Lines for the helium and vacuum pump were connected to both the electrolyte chamber and the receivers. A line leading to a bubbler was connected to the electrolyte chamber. The electrode leads were connected through cables to a selenium rectifier which furnished direct current for electrolysis. The rectifier had a maximum output rating of 250 amp and 6 volts.

The electrolyte chamber was heated by an electric pot furnace with heating elements on all four sides. The furnace was rated at 40 kw and was operated on 230 volts and a three-phase power source. It was built of insulating bricks to form 12-inch-thick walls and a 4-1/2-inch-thick bottom. The outer surfaces of the bricks were surrounded by 2 inches of loosely packed magnesia contained in a thin iron shell. This 61-inch-wide, 56-inch-long, and 32-inch-high furnace was set on a 6-inch channel iron frame.

Figure 4 shows the cell in operation. The cost of the cell and major auxiliary equipment is given in appendix A.

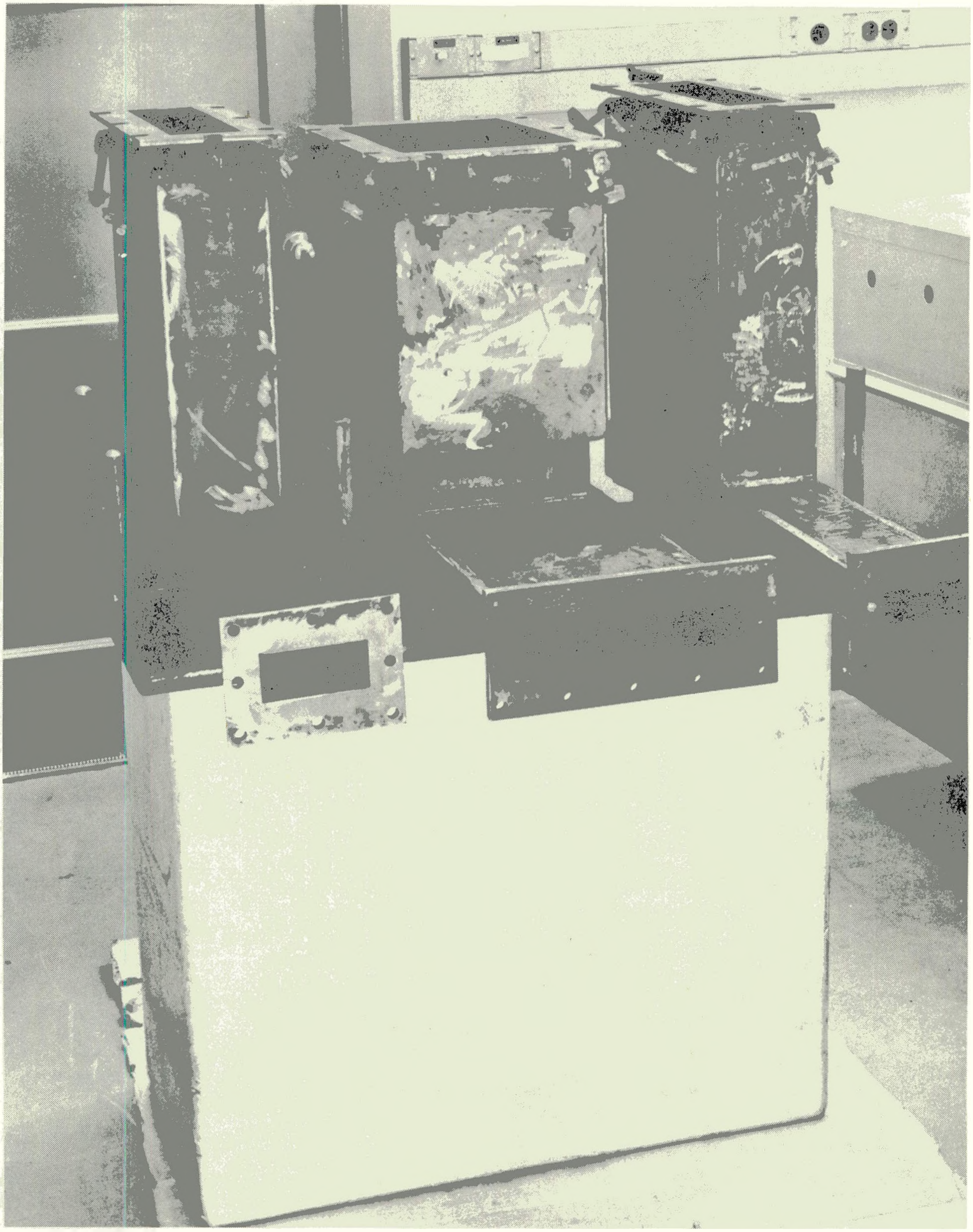

FIGURE 2. - Prototype Cell Before Installation.

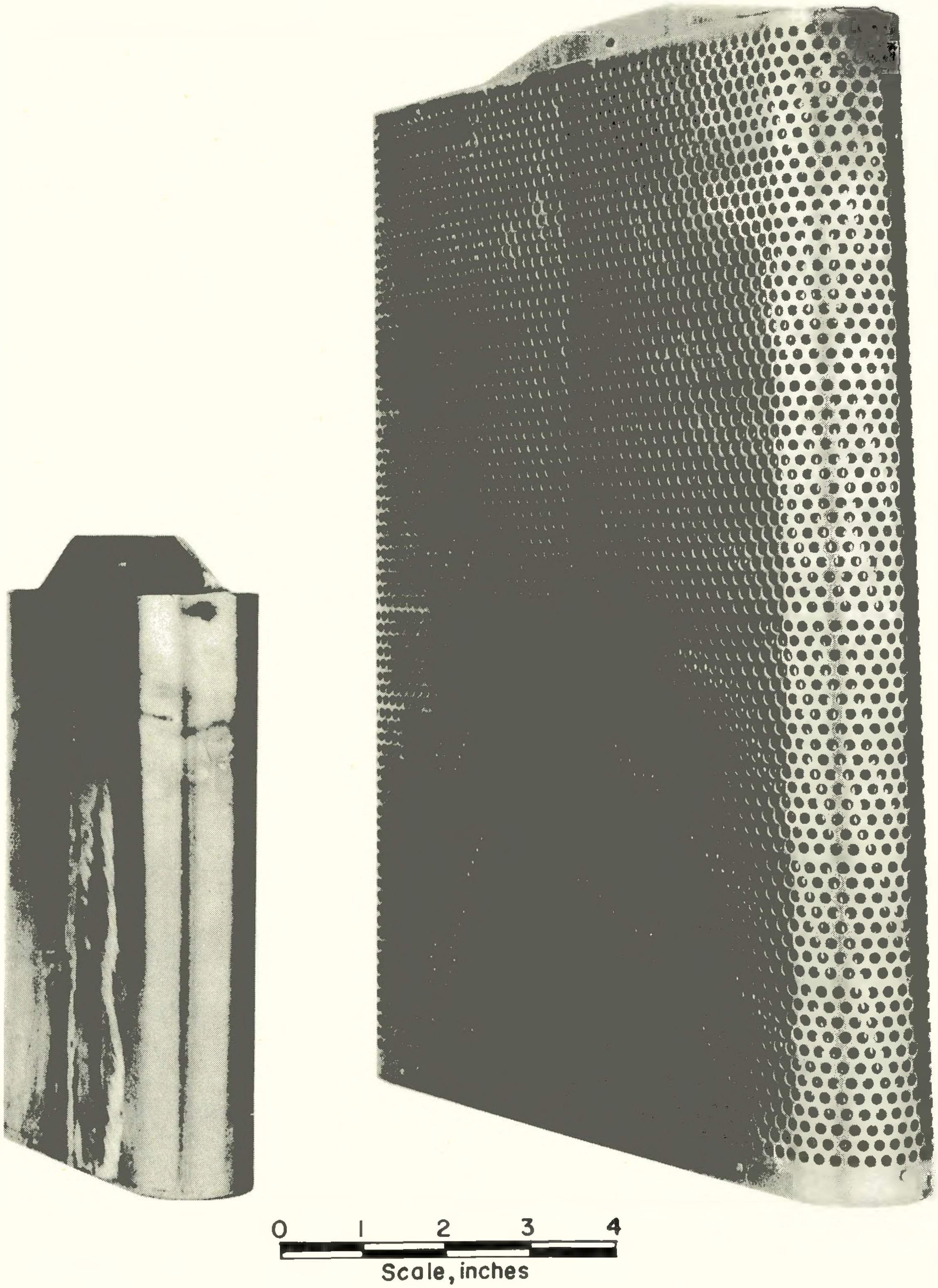

FIGURE 3. - Cathode and Anode Basket.

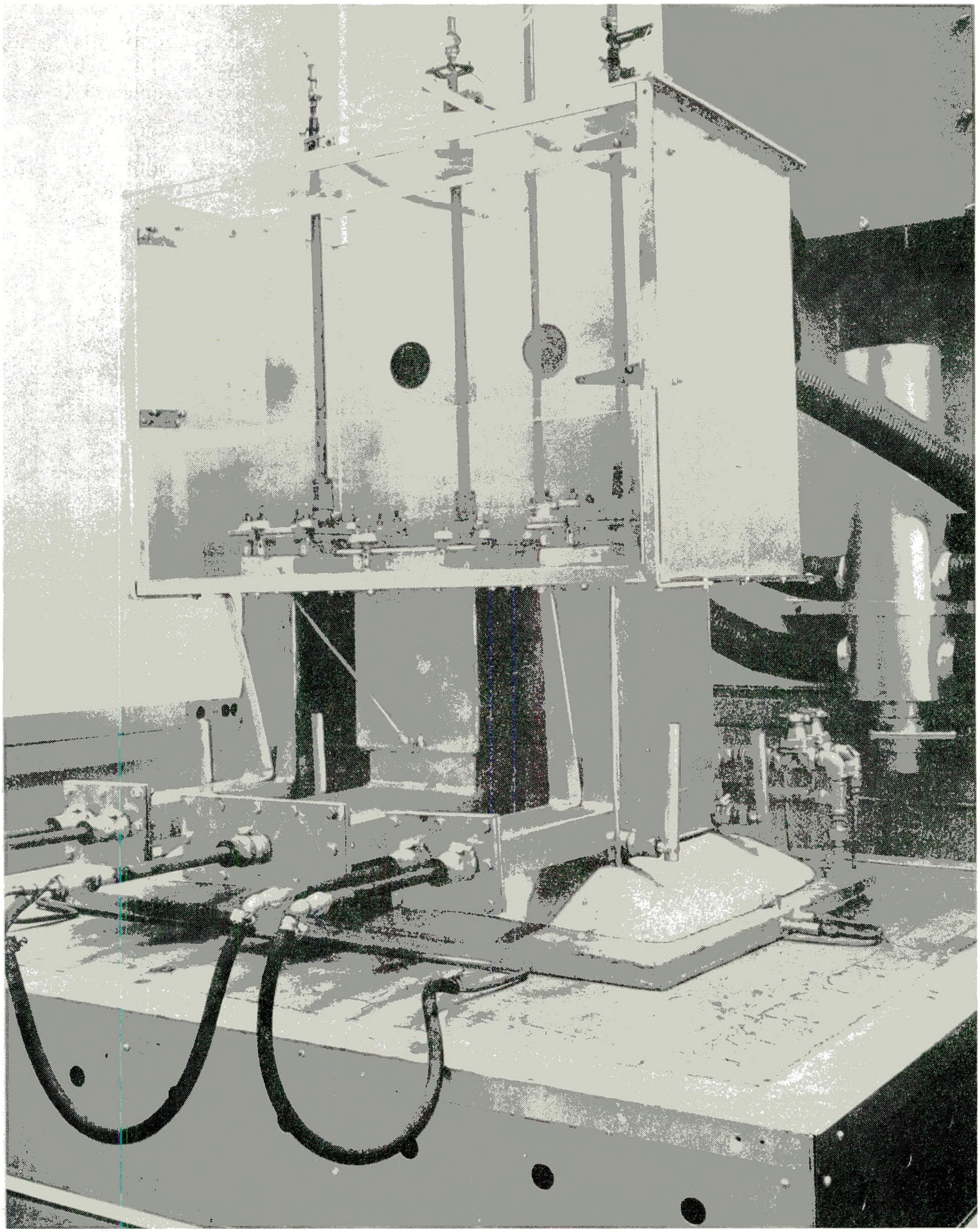

FIGURE 4. - Prototype Cell in Operation.

OPERATING PROCEDURE

After the cell was assembled, it was evacuated to a pressure of 100 μ Hg and then checked for leaks. When the leak rate was less than 5 μ Hg per min with the pump blanked off, the vacuum integrity was considered acceptable and the cell was ready for use.

A mixture of 189.5 lb (86 kg) of chemically pure-grade KCl and 155.5 lb (70.5 kg) of the same grade anhydrous LiCl was added and dried in the cell under vacuum, first at 200° C for 20 hours and then at 250° C for 24 hours. Subsequently the cell was heated to 500° C and filled with helium.

After this point, atmospheric contamination was prevented by closing the slide valves between the electrolyte chamber and the receivers when the receivers were opened to the outside for the introduction or removal of material. The receivers were sealed and purged of air (by evacuating and backfilling with helium three times) before the slide valves were reopened.

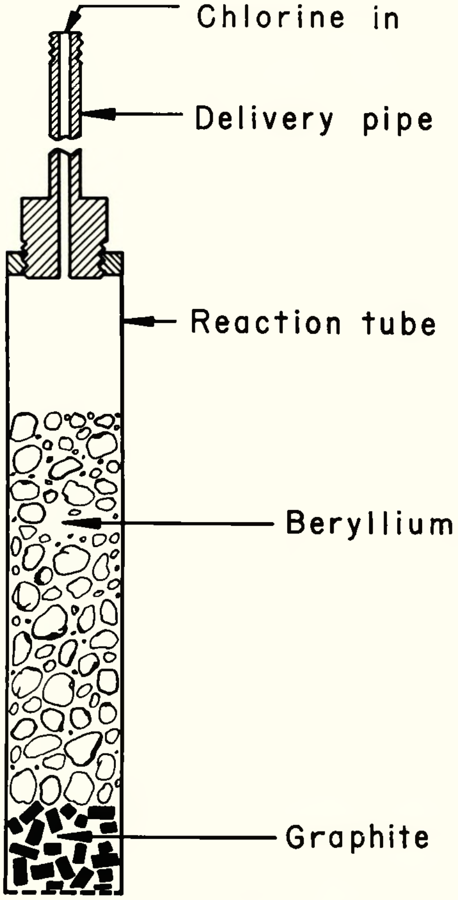

FIGURE 5.-Chloridizing Apparatus.

The $BeCl_2$ component of the electrolyte was produced in situ by reacting chlorine with beryllium metal, $Be + Cl_2 \rightarrow BeCl_2$. The chloridizing apparatus, shown in figure 5 was made of nickel. It consisted of a reaction tube and a delivery pipe. Beryllium metal beads, underlaid with a layer of graphite chunks to prevent subsequent residual material from blocking the passage holes, were charged into the tube. The delivery pipe was slid through an electrode lead port in the cover of a receiver at one end and screwed to the reaction tube at the other. Chlorine was delivered through this pipe to the reaction tube.

During chloridization, the reaction tube was partially immersed in the molten salt. The product $BeCl_2$ passed through the perforated bottom of the reaction tube and was dissolved in the melt. After the stoichiometric amount of chlorine to yield the desired concentration of $BeCl_2$ in the electrolyte was added, the chloridizing apparatus was removed and the electrolyte was sampled and analyzed. (A complete utilization of chlorine was obtained and 24 percent more beryllium than required was provided in the metal charged.) The cost of the electrolyte is detailed in appendix B.

Approximately 2.2 lb (1 kg) of scrap metal chips were loaded in each of the anode baskets which were then immersed 14 inches into the electrolyte. The cathode was immersed 6 inches. Electrolysis was performed with one anode basket on each side of the cathode.

At the end of a deposition cycle, the deposit was raised just above the electrolyte and allowed to drain for 5 minutes. It was subsequently sealed in the receiver and cooled for 40 minutes.

After the deposit was removed from the cell, it was leached in 1-percent HNO_3 solution for 1-1/4 hours, stripped from the cathode, broken into flakes in an electric blender (2 minutes), and stirred in a fresh 1-percent HNO_3 solution for 1-1/2 hours and in distilled water for 1-1/2 hours. Then the deposit was soaked in fresh distilled water for 18 hours, filtered, washed, rinsed with acetone, and dried.

The anode baskets were left in the electrolyte until the metal recovered by electrolysis was equal to approximately 50 percent of the metal charged as the anode feed. When this depletion of the anode material was reached, the baskets and the residual anode metal were removed, completing one cycle of utilization of the anode feed.

ANODE FEED

Beryllium scrap metal chips, shown on the left in figure 6, were used as the anode feed. These chips were generally 7/16 inch wide, 1/64 inch thick, and 1/4 to 1/2 inch long. Chemical analyses of this material (nominally 96 to 97 percent Be) varied somewhat from sample to sample. The average analysis of four samples showed impurities, in parts per million, as follows: O, 9,200; N, 400; C, 600; Ag, 10; Al, 530; Ca, 79; Cr, 155; Cu, 170; Fe, 2,000; Mg, 150; Mn, 135; Mo, 44; Ni, 240; Pb, 110; Si, 450; and Ti, 410.

TESTS AND RESULTS

Optimum Current and Deposition Duration

Major areas programmed for investigation in the prototype cell were (1) the maximum current at which metal of acceptable quality could be produced and (2) the duration of deposition cycle in which a deposit of maximum size could be produced and recovered satisfactorily.

After several deposits were made to clean the electrolyte, two series of tests were conducted to establish the optimum amperage and deposition cycle. The first series consisted of tests made with a 6-1/2-hour deposition period and currents ranging from 50 to 200 amp. The operating data and analyses of the impurities in the products are given in tables 1 and 2, respectively.

Operations at 150 amp or lower were smooth, and the purity of the products was satisfactory. In the 200-amp operation, severe polarization occasionally occurred that required a shaking of the electrodes to restore normal operation, and the product contained an unacceptably high amount of nickel.

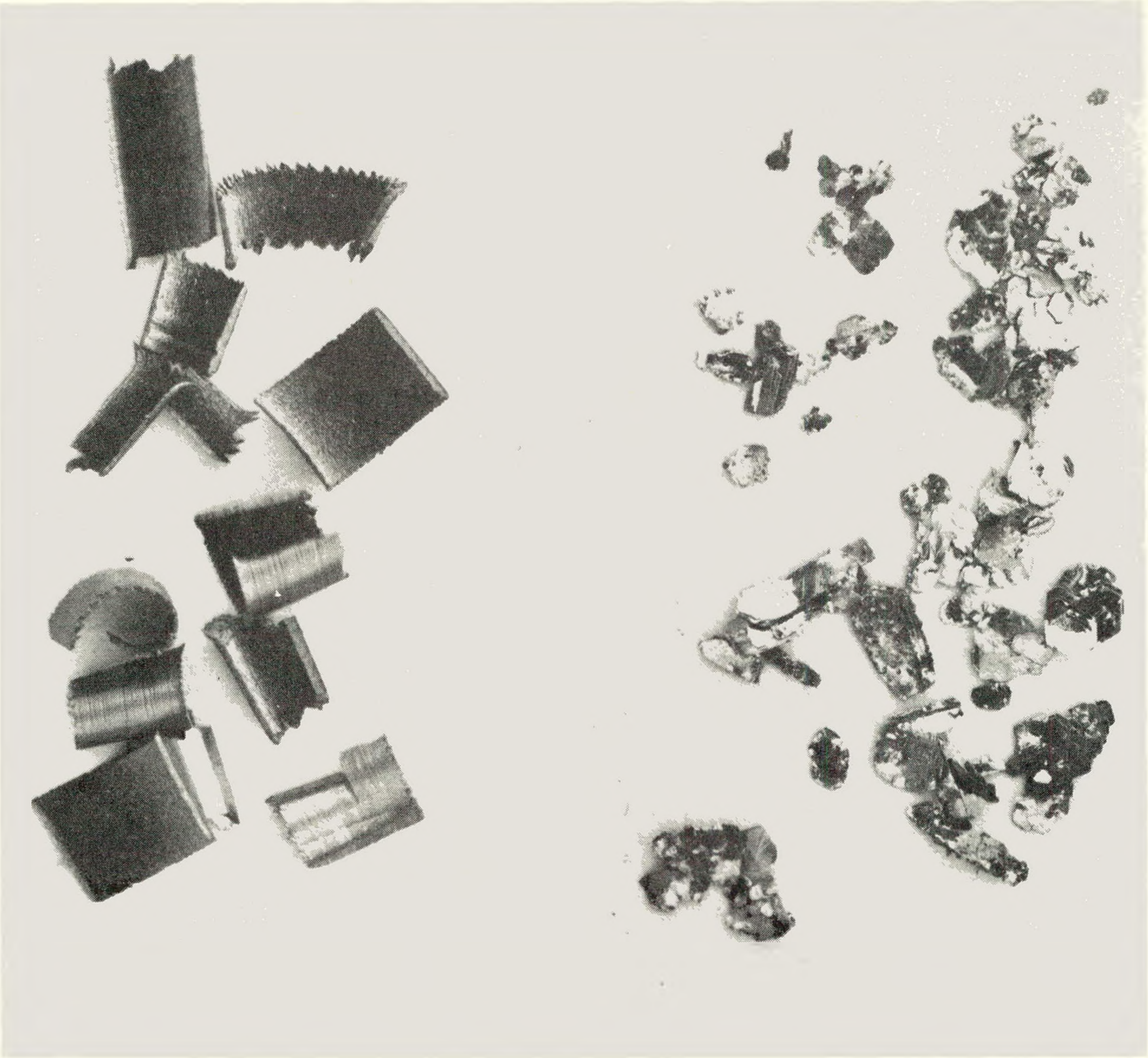

FIGURE 6. - Beryllium Feed and Product (X 4).

TABLE 1. - Cell operation with various amperages[1]

Amperage	Average voltage	Cathode current density, amp per sq ft[2]	Deposit		Cathode current efficiency, percent
			Metal weight, g	Ratio of salt to metal	
50	0.62	94	40.0	2.2	73
100	1.30	189	73.0	3.4	67
150	1.62	283	86.0	3.3	52
200	1.98	378	107.2	4.1	49

[1] Tabulated results are averages of 3 tests.

[2] Current divided by initial cathode area.

TABLE 2. - Products made with various amperages[1]

Amperage	Impurities, ppm[2]								
	Cl	Al	Ca	Cu	Fe	Mg	Mn	Ni	Si
50	300	<50	<50	17	<75	8	<10	<60	<50
100	400	<50	<50	20	<75	9	15	<60	<50
150	400	<50	<50	28	<75	15	21	<60	<50
200	600	<50	<50	26	<75	17	26	>500	<50

[1] Tabulated results are averages of 3 tests.

[2] Other metallic impurities that were not detected include Ag, B, Bi, Co, Cr, Mo, Pb, Sn, Ti, and V.

Tests to establish the optimum deposition duration were made with a current of 150 amp (a cathode current density of 283 amp per sq ft) and deposition durations ranging from 4 to 12 hours. The operating data are shown in table 3, and the analyses of the impurities in the products are given in table 4. All metal products were of acceptable purity.

TABLE 3. - Cell operation with various deposition durations[1]

Duration, hours	Average voltage	Deposit metal weight, g	Average deposition rate, g per hr	Cathode current efficiency, percent
4	1.45	78.7	19.7	78
6-1/2	1.62	86.0	13.2	52
12	1.60	150.6	12.6	50

[1] Tabulated results are averages of 3 tests.

TABLE 4. - Products made with various deposition durations[1]

Duration, hours	Impurities, ppm[2]								
	Cl	Al	Ca	Cu	Fe	Mg	Mn	Ni	Si
4	500	<50	<50	<15	<75	9	<10	<60	<50
6-1/2	400	<50	<50	28	<75	15	21	<60	<50
12	600	<50	<50	16	<75	9	27	<60	<50

[1] Tabulated results are averages of 3 tests.

[2] Other metallic impurities that were not detected include Ag, B, Bi, Co, Cr, Mo, Pb, Sn, Ti, and V.

The biggest deposit was produced in the 12-hour period, but the highest average rate of deposition was obtained with the 4-hour period. As the time and labor expended in cooling the deposit and exchanging cathodes were fixed on a per-deposit basis, the optimum deposition duration was, therefore, the duration in which a deposit of maximum size could be produced and recovered satisfactorily.

Many crystals at the bottom of the cathode from the 12-hour depositions were knocked off when the deposits were being raised and sealed in the receiver. Because the deposit size exceeded the space allowed by the receiver, the height of the receiver was extended by 4-1/2 inches to mitigate this metal recovery problem in the subsequent cell operation.

Production of Metal

A series of tests was made for the production of metal under optimum conditions to demonstrate the process behavior in sustained operation. Certain operating cost data on the production of metal were also obtained.

This series consisted of 12 deposits produced from 2 batches of anode feed (2 baskets in each batch). The current was 150 amp (with a cathode current density of 283 amp per sq ft), and the average cell voltage was 1.33 volt. The deposition duration was 10-3/4 hours. Since it took three-quarters of an hour to terminate the test and cool the deposit and one-half hour to exchange cathodes and start a new test, two deposits could be produced a day.

The extension of the cathode receiver effectively prevented the clipping of crystals from the bottom of the deposit. A deposit as removed from the cell is depicted in figure 7. Its size was about the maximum that the receiver could accommodate.

The metal product, shown on the right in figure 6, averaged 188.1 g (0.415 lb) per deposit, and the average cathode current efficiency was 69 percent. The impurities of the product, in parts per million, were as follows:[7] Cl, 800; C, 700; O, 610; N, <10; K, 440; Li, 60; Al, 34; Ca, 32; Cu, 20; Fe, 25; Mg, 9; Mn, 23; Ni, <60; and Si, <50.

A total of 2,257 g (approximately 4.98 lb) of metal was produced from the starting anode feed of 4,411 g (approximately 9.72 lb), representing a recovery of 51 percent. The remaining anode feed was withdrawn, but could be made usable for recycling after an acid leaching (described in the following section).

Certain operating cost data for electrorefining beryllium are itemized in appendix C.

Recycling of Anode Feed

To maintain control over the purity of the deposit metal, the anode feed had to be replaced after about 50-percent depletion. Recycling of anode feed was tested. After 50-percent depletion, the anode material was removed from the cell, leached in 5-percent nitric acid solution, and dried; then the minus 8-mesh particles were removed. The tests were performed with a current of 100 amp (a cathode current density of 189 amp per sq ft) and a deposition duration of 7-1/2 hours. The operating data and analyses of the product of these anode-recycling tests are compared in tables 5 and 6 with those of tests made in identical conditions, using fresh anode feed.

---

[7] The lower determination limits for Al, Ca, and Fe were extended, as refinements in the analytical procedure were made. Melting the product under an inert atmosphere reduced the Cl content to less than the analytical determination limit of 100 ppm, the K content to about 175 ppm, and the Li content to less than the analytical determination limit of 10 ppm.

FIGURE 7. - Deposit as Removed From Cell.

TABLE 5. - A comparison of cell operations using fresh anode feed and recycled anode feed[1]

Anode feed	Average voltage	Deposit		Cathode current efficiency, percent
		Metal weight, g	Ratio of salt to metal	
Fresh................	1.03	96.2	2.6	76
Recycled.............	1.10	112.8	2.7	89

[1] Tabulated results are averages of 11 tests.

TABLE 6. - A comparison of products electrodeposited from fresh anode feed and recycled anode feed[1]

Anode feed	Impurities in products, ppm[2]										
	Cl	O	H	C	Al	Ca	Cu	Fe	Mg	Mn	Si
Fresh....................	400	660	30	250	<20	34	20	27	8	<10	<50
Recycled.................	700	1,060	40	130	67	55	<15	81	19	16	79

[1] Tabulated results are averages of 11 tests.

[2] Other metallic impurities that were not detected or below the determination limit include Ag, B, Bi, Co, Cr, Mo, Ni, Pb, Sn, Ti, and V.

The recycled anode feed produced somewhat smaller crystals and yielded a higher cathode current efficiency than did the fresh anode feed. The higher cathode current efficiency was chiefly attributed to more complete recovery of crystals because the small crystals were generally less subject to mechanical loss than the big ones.

Metal produced from the recycled anode feed contained higher impurities than did the product made from the fresh anode feed. The product made from the recycled anode feed, however, still showed a high degree of refining compared with the original anode feed.

DISCUSSION

Metal produced from the prototype cell was similar in appearance and in purity to that produced from laboratory-scale cells (12 inches in diameter and 30 inches long with 60 lb of electrolyte).[8] The cathode current efficiency was somewhat reduced because of the increased mechanical loss incurred by the bigger deposit produced in the prototype cell. The optimum current was increased from 35 amp in the laboratory-scale cells to 150 amp in the prototype cell. In most of the other aspects of the cell performance, there was little difference attributable to the scaling-up of the cell.

In 2 years of continuous operation of the prototype cell, no major mechanical difficulty was experienced. The $BeCl_2$ concentration remained substantially constant, indicating little or no volatilization of $BeCl_2$ from the electrolyte. The electrolyte, however, had to be replenished to its original volume whenever 20 to 25 percent of it was consumed (caused mainly by dragouts with the cathode deposits and the anode residues) in order to continue operation at the cathode immersion for which the cell was designed.

---

[8] First work cited in footnote 3.

With periodic electrolyte replenishments, impurities in the electrolyte did not increase. This is illustrated in table 7. The absence of impurity buildup in the electrolyte was also corroborated by the constant level of purity of the deposit metal maintained throughout the 2 years of operation.

TABLE 7. - Impurities in the electrolyte

Electrolyte sample[1]	Analysis, ppm									
	Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Si	Ti
Before deposit 1.........	300	<100	<10	<5	200	400	<100	<100	<100	<20
After deposit 48.........	300	<100	<10	<5	150	500	<100	<100	<100	20
After deposit 78.........	300	<100	<10	<5	100	600	<100	<100	<100	20
After deposit 331........	200	100	<10	<5	100	400	<100	<100	100	<20

[1] The first 3 electrolyte samples were taken in the first, middle, and last parts of the fresh electrolyte period before any electrolyte replenishment was made. The last electrolyte sample (after deposit 331) was taken just before the cell operation was terminated.

CONCLUSIONS

Operation in the prototype cell demonstrated that adapting the beryllium electrorefining process from small, laboratory-scale cells to the relatively large, prototype cell presented few difficulties in mechanical functions, maintenance of optimum operating conditions, and control of product purity. Enlarging the scale of operation did not result in any attendant lowering of the purity of the product.

The prototype cell was in continuous operation for 2 years. With periodic electrolyte replenishments, no deterioration in performance was observed, and no major repair of the cell was required.

Recycling of the spent anode feed was also tested. The spent anode material was suitable for recycling after it had been leached in a 5-percent nitric acid solution and the fine-sized particles were screened off. The metal produced from the recycled anode feed, however, contained somewhat higher impurities than that produced from the fresh anode feed.

APPENDIX

TABLE A-1. - Cost of prototype cell and major auxiliary equipment

Item:[1]	Cost
1 each, mild steel electrolyte chamber and cover assembly	\$730.00
1 nickel cell liner	257.60
2 nickel anode baskets and 1 cathode	123.37
3 nickel electrode leads	95.94
1 electric furnace	650.00
1 selenium rectifier	350.00
1 vacuum pump	262.50

[1] See the equipment section for detailed description.

TABLE A-2. - Cost of electrolyte[1] [2]

Item:		Cost
Labor (actual working time):		
Mixing, loading, and drying LiCl and KCl...man-hours	16	
Reacting Be metal with Cl_2...do...	98	
Total...do...	114	
114 man-hours at \$3.06/man-hour		\$348.84
Electricity (heating cell):		
Drying LiCl and KCl and heating to 500° C...kwhr	658	
Reacting Be metal with Cl_2...kwhr	628	
Total...kwhr	1,286	
1,286 kwhr at \$0.005/kwhr		6.43
Water (cooling cell parts):		
Drying LiCl and KCl and heating to 500° C...gal	47,300	
Reacting Be metal with Cl_2...gal	32,640	
Total...gal	79,940	
79,940 gal at \$0.11/1,000 gal		8.79
Helium (cell inert atmosphere):		
Reacting Be metal with Cl_2...cu ft	149	
149 cu ft at \$0.043/ cu ft		6.41
Materials:		
KCl, 189.5 lb at \$0.40/lb	\$75.80	
LiCl, 155.5 lb at \$0.95/lb	147.73	
Cl_2, 56.0 lb at \$0.15/lb	8.40	
Be metal,[3] 7.34 lb at \$50/lb	367.00	
Total cost of materials		598.93
Total cost for 408.1 lb of electrolyte		969.40
Cost per pound of electrolyte (\$969.40/408.1 lb)		2.38

[1] On the basis of preparing 408.1 lb of electrolyte with the composition of 52.4 mole-percent LiCl, 36.3 mole-percent KCl, and 11.3 mole-percent $BeCl_2$.

[2] Not including capital, overhead, and supervision costs.

[3] Beryllium beads, 97 percent pure.

TABLE A-3. - Operating cost per pound of electrorefined beryllium[1]

Item:			Cost
Labor (actual working time):			
Operating cell	man-hours	1.93	
Leaching product[2]	do	2.49	
Repairs, clean up, etc	do	.22	
Total	do	4.64	
4.64 man-hours at $3.06/man-hour			$14.20
Electrolyte dragout:			
With cathode	lb	1.908	
With anode residue	lb	1.636	
Total	lb	3.544	
3.544 lb at $2.38/lb			8.43
Electricity:			
Heating cell	kwhr	226.4	
Electrolysis	kwhr	6.8	
Total	kwhr	233.2	
233.2 kwhr at $0.005/kwhr			1.17
Water (cooling cell parts): 13,660 gal at $0.11/1,000 gal			1.50
Helium: 10.6 cu ft at $10.4/240-cu-ft cylinder			.46
Materials for leaching and cleaning:			
Nitric acid, 1.053 lb at $0.20/lb		$0.21	
Demineralized water, 15.9 gal at $20/1,000 gal		.32	
Acetone, 0.733 lb at $0.36/lb		.26	
Hydrochloric acid,[3] 0.471 lb at $0.173/lb		.08	
Total cost of materials			.87
Operating cost per pound of electrorefined beryllium metal			26.63

[1] Not including capital, overhead, supervision, and anode feed costs.

[2] Based on leaching product in 0.415-lb (1 deposit) batches. Cost for this item can be greatly reduced in a large-scale operation.

[3] Used in cleaning cathodes and anode baskets.