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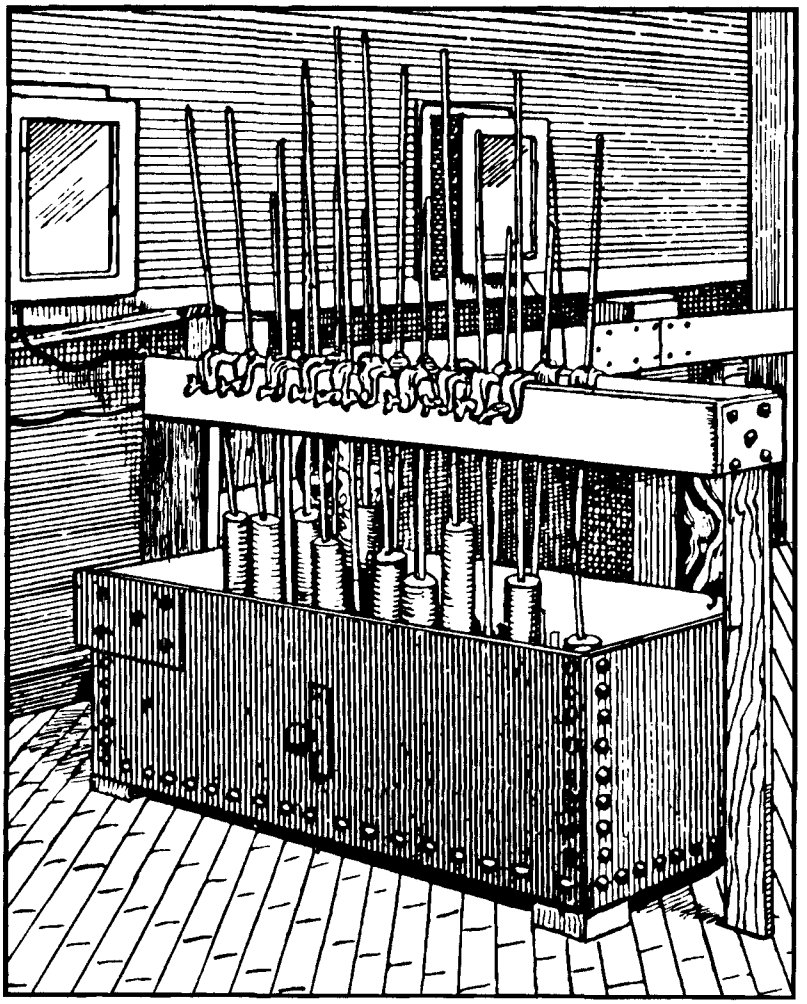
Inert Anode/Cathode Program

Development of a Standard Bench-Scale Cell for Electrochemical Studies on Inert Anodes

July 1986



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On the cover:

Aluminum reduction pots at the Pittsburgh Reduction Company's (Alcoa's) plant in 1889. Adapted from a photograph, courtesy of Alcoa.

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Inert Anode/Cathode Program

DEVELOPMENT OF A STANDARD BENCH-SCALE CELL FOR ELECTROCHEMICAL STUDIES ON INERT ANODES

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ABSTRACT

The objective of this work was to develop a standard bench-scale cell for performing short-term ac and dc polarization studies on inert anode candidate materials in molten cryolite.

Two designs for electrochemical cells were developed and successfully evaluated in short-term experiments. Both cells consisted of the inert anode as a small cylindrical specimen partially sheathed in alumina, an Al/Al_2O_3 reference electrode, and a cryolite bath saturated in alumina. The difference between the two cells was in the design of the cathode. One cell used a bare solid metal cathode; the other used an aluminum pad similar to the Hall-Heroult configuration.

CONTENTS

FIGURES

INTRODUCTION

The Inert Anode/Cathode Program is conducted by Pacific Northwest Laboratory[a] for the U.S. Department of Energy (DOE), Office of Industrial Programs (OIP). The purpose of the program is to develop long-lasting, energy-efficient anodes, cathodes, and ancillary equipment for Hall-Heroult cells used by the aluminum industry. The program is divided into four tasks:

- Inert Anode Development - to improve the energy efficiency of Hall-Heroult cells by development of inert anodes.
- Cathode Materials Evaluation - to confirm the chemical behavior of carbon-containing TiB_2 cathode material (TiBCM) in the presence of molten cryolite and aluminum, and to upgrade the analytical procedures for Al_4C_3 in cryolite.
- Cathode Bonding Development - to develop methods for retrofitting Hall-Heroult cells with TiB_2-based cathode materials.
- Sensor Development - to devise sensors to control the chemistry of Hall-Heroult cells using stable anodes and cathodes.

The objectives of the Electrochemical Evaluation Subtask of the Inert Anode/Cathode Program are to develop new candidate materials for inert anodes for use by the aluminum industry and to determine and broaden the operating range of these anodes. Toward this end, bench-scale electrochemical and in situ laser Raman spectroscopic studies will be used to identify the reaction mechanisms for anodes in molten cryolite.

Prior to performing the electrochemical evaluation of inert anodes, a suitable cell for polarization studies was required. It is important that the electrochemical cell lend itself to a "standard test." Few, if any, operating characteristics of the cell should be variable, unpredictable, or unknown. Use of a standard cell in conjunction with standard experimental procedures permits

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valid performance comparisons between the different inert anode candidate materials. Two successful designs for electrochemical cells were developed in this work and are shown in Figure 1.

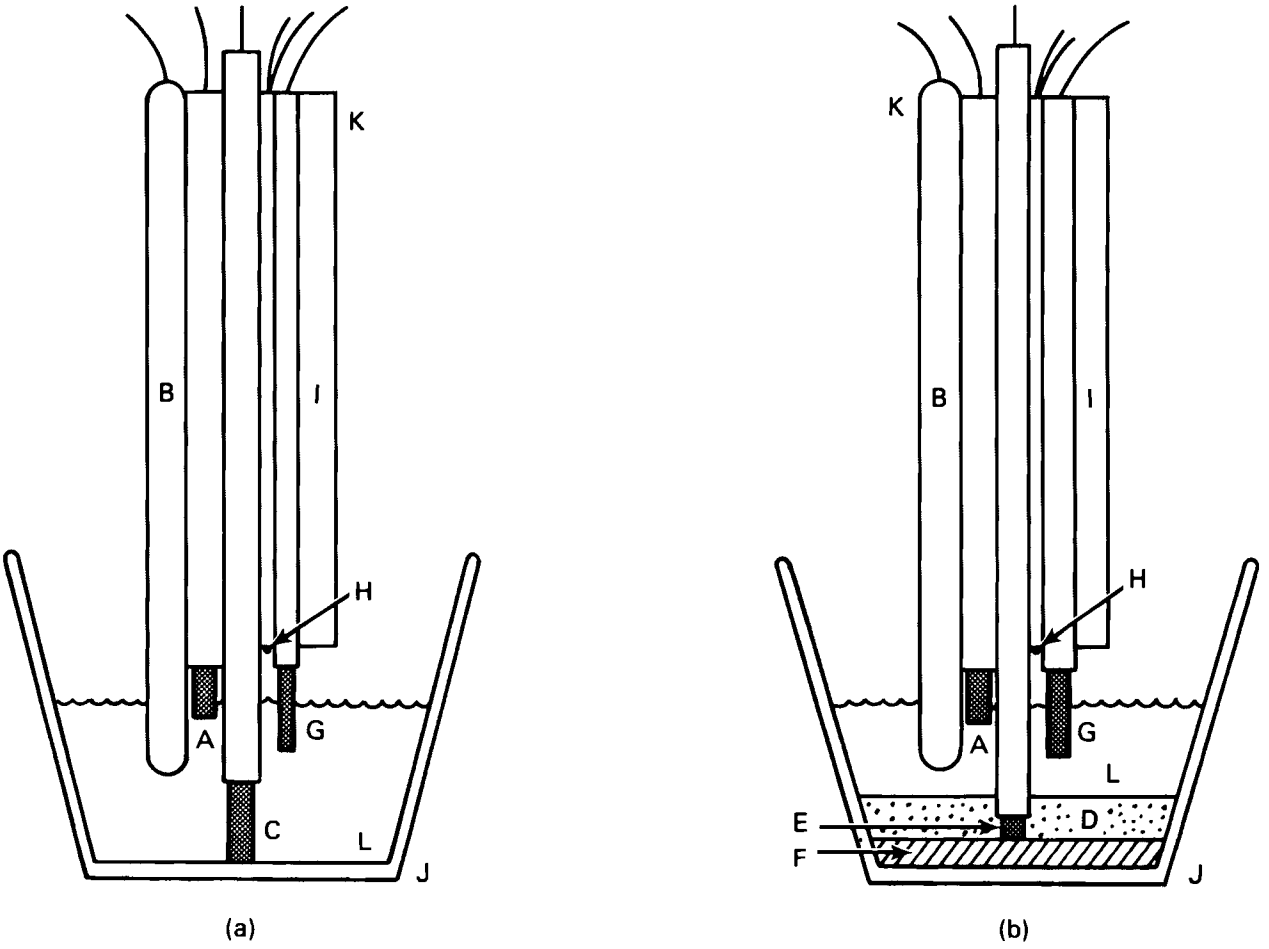

FIGURE 1. Two Successful Electrochemical Cells Using (a) a Solid Metal Cathode and (b) an Aluminum Pad Cathode. Cell components: (A) inert anode, (B) reference electrode, (C) solid metal cathode, (D) aluminum pad, (E) solid metal lead to aluminum pad, (F) TiB_2 substrate, (G) platinum control anode, (H) thermocouple, (I) gas purge tube, (J) alumina crucible, (K) alumina sheathing, and (L) cryolite.

CONCLUSIONS

Two successful designs for short-term electrochemical polarization studies on inert anodes were developed. The designs differ only in their cathode configuration. The solid metal (preferably tungsten) cathode should be used for rapid short-term tests when the presence of significant amounts of aluminum metal is undesired or unnecessary. The aluminum cathode should be used when conditions closer to an operating Hall-Heroult cell are required.

Future tests will use one of the two cell designs developed in this work to evaluate inert anode candidate materials. Despite the proven reliability of these two configurations, some minor modifications may still be made. In particular, since a more durable cathode is desired, investigations in this area will continue.

DISCUSSION

Each candidate cell design was investigated using the Alcoa 5324 inert anode--an $NiFe_2O_4$-based ceramic material. The electronic equipment used in these experiments (essentially the PAR Model 368 ac Impedance System) is shown in Figure 2. For the inert anode in each cell, dc polarization curves were obtained. In some cases, ac impedance data were also collected. The quality of the polarization data, the reproducibility of these data, and the integrity of the cell during operation were the principal criteria used in the evaluation.

The initial cell design investigated was based on preliminary experimental work done at PNL and on similar experiments performed at Aluminum Company of

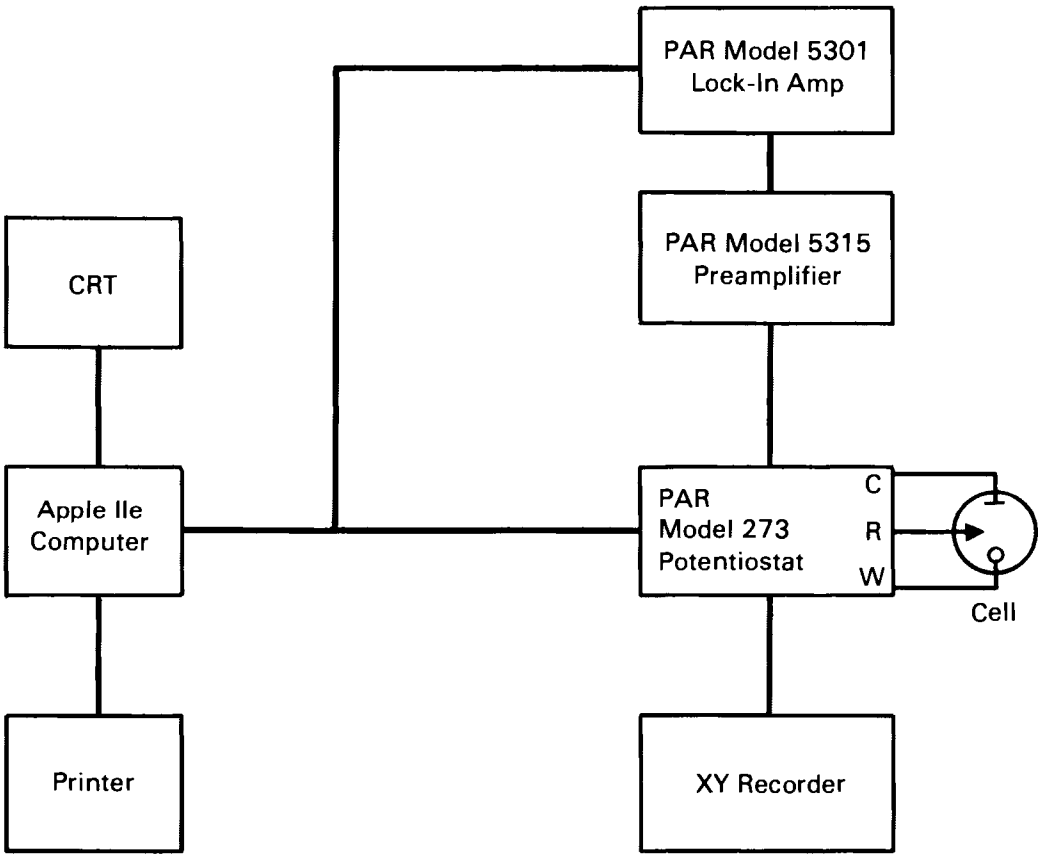

FIGURE 2. PAR Model 368 ac Impedance System

America (Alcoa) Laboratories (Weyand et al. 1985). Alcoa used a three-electrode configuration: the inert anode, a molybdenum cathode, and an Al/Al_2O_3 reference electrode. This electrode configuration and variations of it were evaluated in our work. The cell vessel was made of alumina. The electrolyte composition (designated GM#2) was approximately 72% natural Greenland cryolite (Na_3AlF_6), 12% AlF_3, 12% Al_2O_3 (saturated), and 4% CaF_2. The weight ratio of NaF to AlF_3 (bath ratio) was 1.1:1. The bath was saturated in alumina to minimize dissolution of the cell container and alumina-sheathed cell components. Experiments were conducted at temperatures between 960°C and 1040°C.

The following components of the electrochemical cell were investigated.

INERT ANODE

The principal inert anode material used in all the cell development work was the Alcoa 5324 composition. (A better "control" anode may be warranted as described in the next section.) The most favorable arrangement was to sheath the electrode in alumina (at least partially) and to submerge it only about 1 mm in the molten cryolite.

The inert electrodes were cylindrical specimens about 1.6 cm in length with a terminal area of 0.16 to 0.17 cm^2. Each specimen was sheathed in an alumina tube whose inner diameter was such that there was an extremely close fit between the specimen and the tube. As shown in Figure 3, two sheathing configurations were used. In the design shown in Figure 3a, only the terminal area of the specimen was left unsheathed for exposure to the molten bath. However, scanning electron microscope/energy-dispersive x-ray (SEM/EDX) analysis of cross sections of the specimens (which had been immersed in the molten salt for up to 6 h) showed that, for this design, there was significant penetration of electrolyte into the very narrow gap between the electrode and the alumina tube. The penetration occurred even though the anode and the alumina tube showed very little deterioration.

Creep of the electrolyte into the gap between the anode and the sheathing material may introduce significant uncertainty (about 20% for a 0.25-mm penetration depth) about the specimen's exposed surface area as well as the current density measurements. To circumvent this problem, an alternate anode design

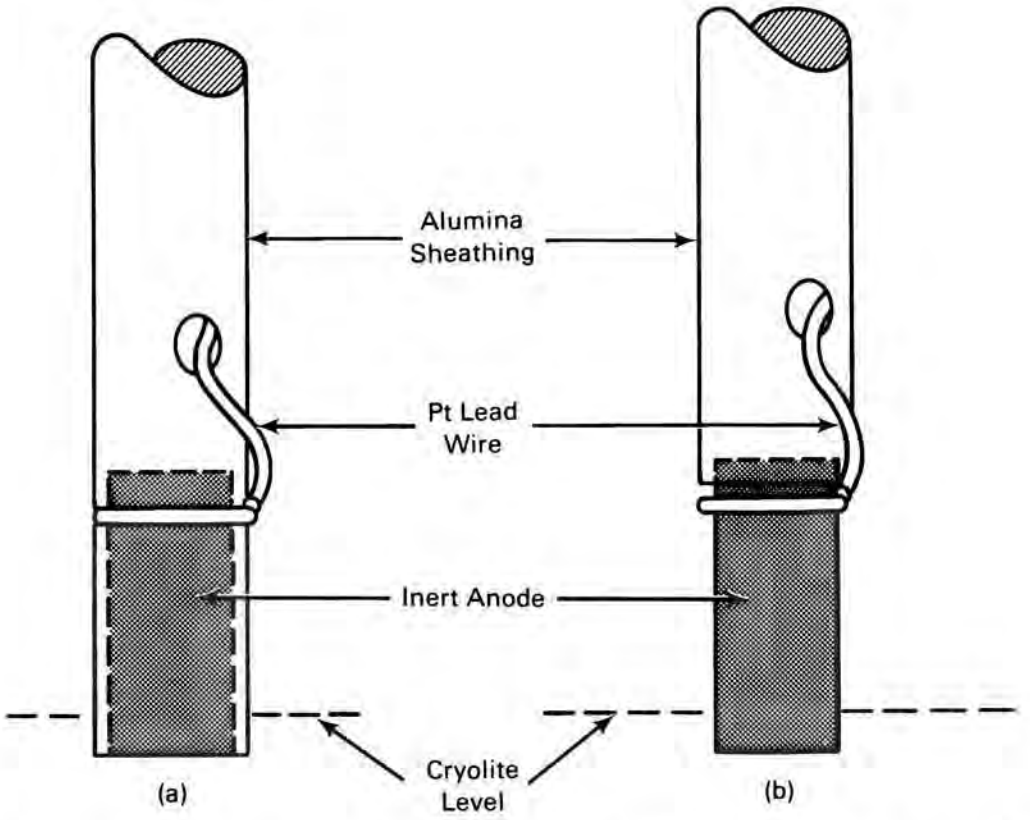

FIGURE 3. Anode Configurations Using (a) Alumina Sheathing Right to the End of the Anode and (b) Alumina Sheathing Retracted from the End of the Anode

was used. As shown in Figure 3b, this design involved retracting the alumina sheath a significant distance away from the end of the anode material. The anode was then submerged in the molten cryolite so that only the terminal area and a very small amount of the circumferential surface were wet. While the total exposed anode surface area was unknown during the experiment, it could be determined afterwards by examining the electrode. The advantage of this design was that it prevented capillary action (present in the previous design) from aggravating the cryolite creep phenomenon. The principal limitation was that the exposed surface area was not known exactly until the end of the experiment.

Since the cell did not allow for visual monitoring of the electrodes, the extent to which the anode assembly was submerged in the molten cryolite was estimated indirectly using alumina rods. The rods were dipped in the molten

bath to gauge its depth. This value was referenced against the length of the electrode body. This approach was found to be successful.

Another procedure to detect submersion of the anode was also evaluated. In this method, an electrical resistance measurement was made between the anode and a second electrode. It was thought that a sudden decrease in resistance as the electrodes were lowered into the electrolyte would indicate the anode had contacted the molten salt. Unfortunately, false readings (indicating the probes were inserted when they were not) were repeatedly obtained with this method. Apparently, the cryolite vapors condensed on the electrodes, providing an electrode-to-electrode conductive "bridge" prior to their insertion into the melt. For this reason, the electrical approach to determining electrode submersion was abandoned in favor of the mechanical method.

In all cases, the electrical lead to the anode was platinum wire. It was channeled through the alumina sheathing tube to make contact with the anode specimen at a point significantly above the level of the cryolite bath. This design successfully minimized the possibility of an electrical short via electrolyte contact with the lead wire.

CONTROL ANODE

Recent polarization studies performed at PNL on the Alcoa 5324 composition have shown that the material may corrode under some conditions. For this reason, it was considered desirable to have a more "inert" material available in each experiment as a benchmark or control for comparing the polarization behavior of new potential anodes. (A control electrode is useful in differentiating the effects of bath composition, cell geometry, etc., from properties of the test anode itself.) Platinum was evaluated for this purpose and found to be acceptable.

In a typical electrochemical evaluation, a polarization profile for the platinum anode would be obtained prior to and/or subsequent to that of the inert anode being evaluated. Both electrodes would be tested in the same cell and under the same conditions. Results for the platinum electrode would then aid the interpretation of nonideal effects observed for the test specimen.

CATHODE

The principal difficulty in the design of a suitable electrochemical cell has been the development of an adequate cathode material and/or configuration. Despite being able to obtain reliable polarization data for the anode, the original cathode physically deteriorated in molten cryolite during polarization. This failure was deemed important at the time and numerous other cathode materials and configurations were evaluated. Unfortunately, most of these also exhibited poor durability. Various modes of cathode failures are possible as discussed below. The principal assessment is that some cathode deterioration can occur in short-term tests without affecting the polarization properties of the anode.

The original design by Alcoa, a coiled molybdenum wire, was successfully used at PNL to obtain polarization data on the Alcoa 5324 anode. However, this cathode showed signs of mechanical failure after only 2 h of testing. To reduce the extent of deterioration, molybdenum was employed as a cathode in various other configurations, including molybdenum wire of different diameters, molybdenum wire protected and completely enclosed in an alumina sheath, and a molybdenum plug sheathed in alumina with one large flat area exposed to the electrolyte medium. In most cases, polarization data were successfully collected. However, post-test analysis of cathode samples revealed significant interaction between molybdenum and molten cryolite after only 2 h of contact. Complete encasement of the molybdenum cathode in alumina was unsuccessful since it disrupted ion transport, causing the potentiostat to overload. SEM/EDX analyses of the molybdenum cathodes showed significant corrosion when exposed to cryolite for about 40 min. X-ray diffraction analysis indicated that one of the principal corrosion products was MoO_2.

Other solid metal electrode materials were also investigated as potential cathodes, including tantalum, platinum, and tungsten. The materials were tested sequentially in the same electrochemical cell using the electrode assembly shown in Figure 4. The assembly contained a large-diameter alumina tube through which different cathodes could be inserted. Each material was tested in cryolite for about 40 min. Polarization data were successfully collected using all of the cathode materials; however, post-test analyses showed that

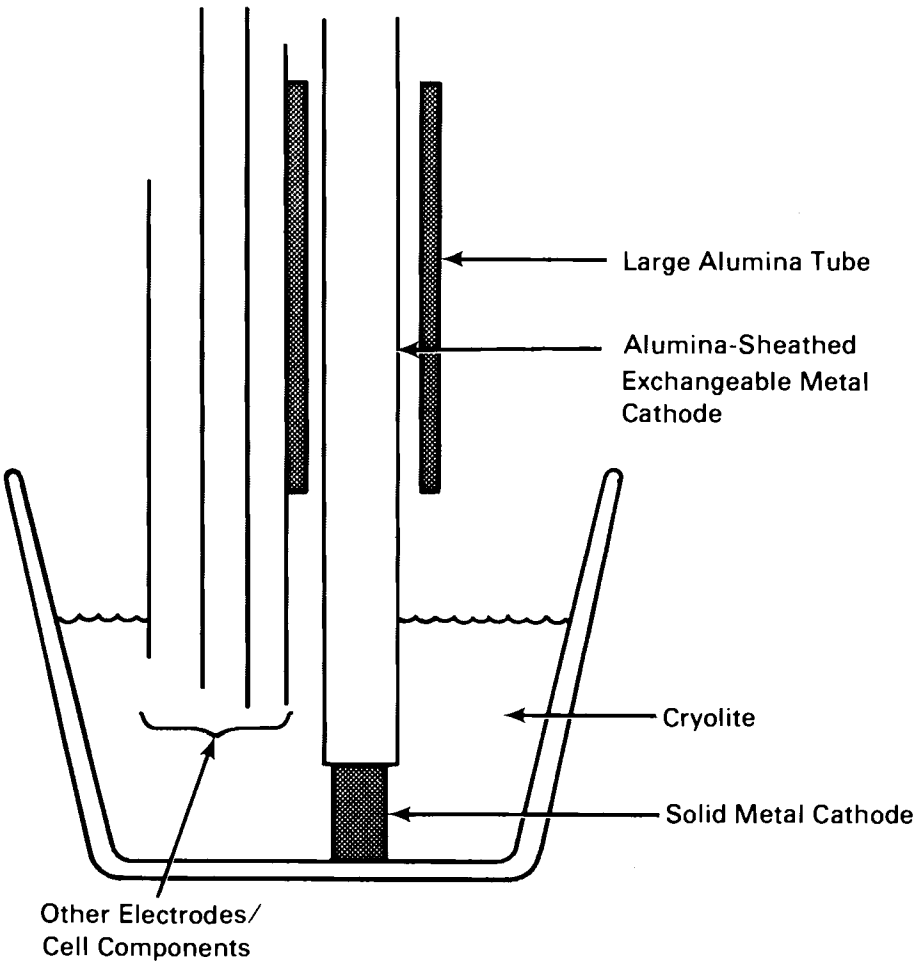

FIGURE 4. Configuration for Testing Solid Metal Cathodes. The large alumina tube facilitated the exchange and repositioning of cathodes during a single experiment.

significant cryolite/cathode interaction occurred in most cases and that the extent of this reaction was different for each metal. Tantalum corroded similarly to molybdenum as revealed by microscopic analysis. The platinum cathode also corroded; but SEM/EDX analysis revealed the presence of rhodium, particularly near the corrosion sites. This finding suggests that a less-than-pure grade of platinum was used in this study and that the failure mode was dissolution of the minor metallic component. Pure platinum is expected to give better results. Tungsten gave the best performance of all the solid metal cathodes tested in the 40-min tests. SEM/EDX analysis indicated very little corrosion. No corrosion products could be detected on the tungsten surface by

x-ray diffraction. More recent studies, however, show that for electrolysis over longer time intervals (2 to 3 h) significant corrosion of tungsten can occur. This corrosion may involve reaction with oxygen generated (in larger quantities in longer term tests) at the anode.

Graphite was also evaluated as an alternative cathode material. However, it also reacted, particularly near the gas phase interface (i.e., above the cryolite melt) in the vicinity of the point of contact between the platinum lead and the graphite cathode. Residual oxygen gas is believed to have caused the reaction. This reactivity was observed despite measures taken to purify the argon purge gas with getters, which reduced levels of oxygen and water vapor to about 1 ppm. Because of the debilitating effects of oxygen on graphite at these temperatures, it is recommended that graphite not be used as the cathode material unless better control of vapor composition is achieved (using a closed or more efficiently purged system).

Other cathode designs investigated in this work were based on the Hall-Heroult molten aluminum cathode. As shown in Figure 5, for one modification, molten aluminum was placed in the bottom of the electrochemical cell and allowed to wet a TiB_2 substrate, which had been attached to the bottom of the alumina crucible using Sauereisen Electrotemp Cement No. 8.[(a)] Electrical contact with the aluminum was made through a metal lead. The lead was partially sheathed in alumina to protect it from the molten cryolite. Molybdenum and tungsten were evaluated as electrical lead materials. Both corroded over a 2-h period with molybdenum showing the most severe attack. Tungsten was more successful but still showed a "thinning" effect probably due to a limited solubility in molten aluminum.

A variation of the Hall-Heroult cathode, using the molten aluminum cathode in a probe-type arrangement, was also investigated (Figure 5b). The utility of the probe design is that it would be reusable, unlike the aluminum "pad," which would have to be prepared anew for each cell. Unfortunately, this design failed, apparently due to plugging of the conduction hole that was required for charge transport across the electrode body boundary.

(a) Sauereisen Cement Co., Pittsburgh, Pennsylvania.

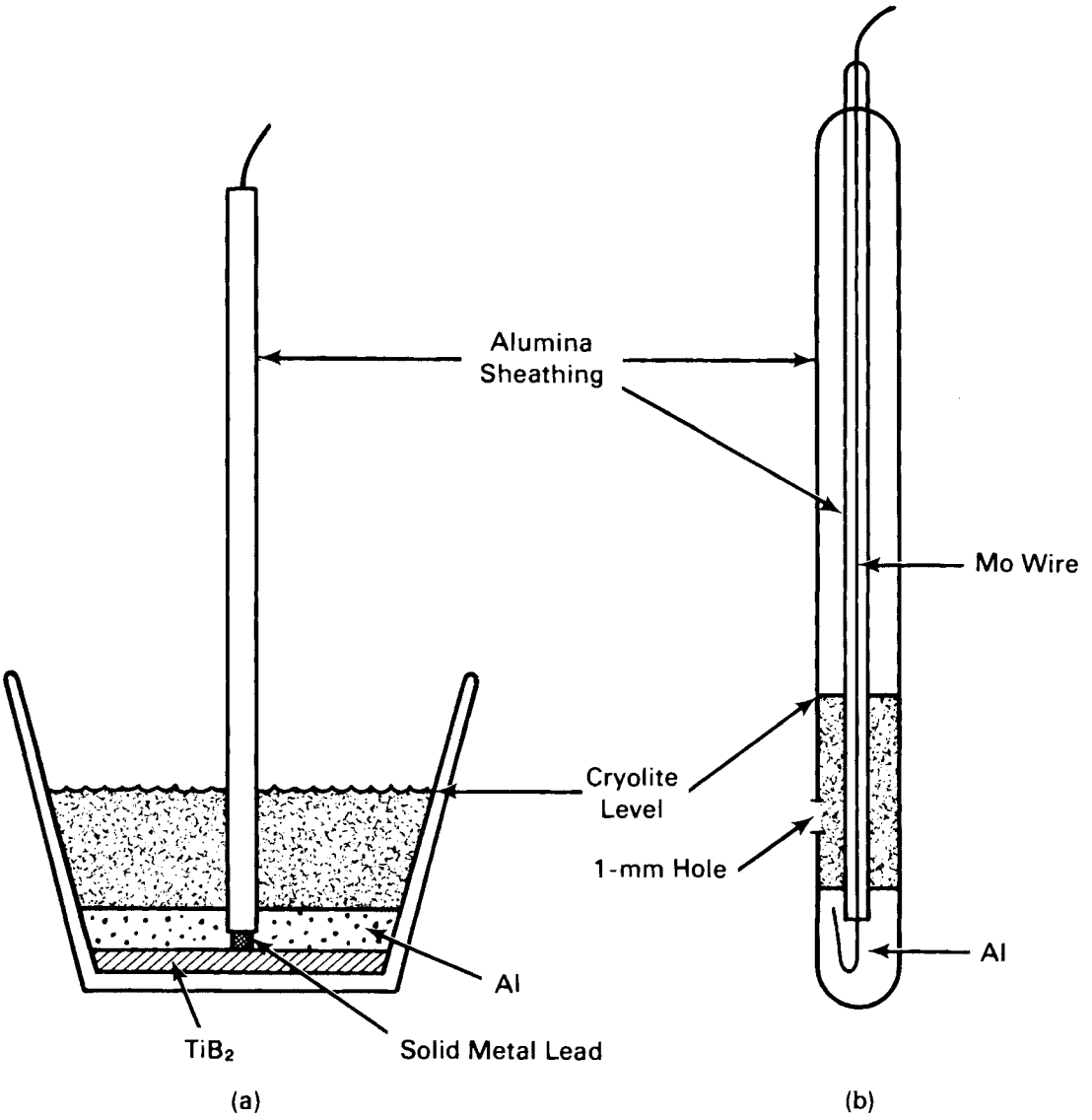

FIGURE 5. Molten Aluminum Cathodes Using (a) an Aluminum Pad Configuration and (b) a Probe-Type Arrangement

In summary, of all of the cathode materials and configurations tested, two designs were found to give the best results. The first design uses a solid metal cathode, preferably tungsten. (A pure grade of platinum may also be suitable.) This cathode is recommended 1) for its ease of assembly and 2) in experiments where minimal dissolved aluminum is required. The second design uses an aluminum pad for experiments where a closer simulation of the Hall-Heroult cell is necessary. Both designs are illustrated in Figure 1.

REFERENCE ELECTRODE

The reference electrode used in this work was developed by Burgman, Leistra, and Sides (1986) and is shown in Figure 6. This electrode was found to function adequately and was the only reference design used. It remained in good condition and was reusable after 4 h of contact with molten cryolite saturated in alumina.

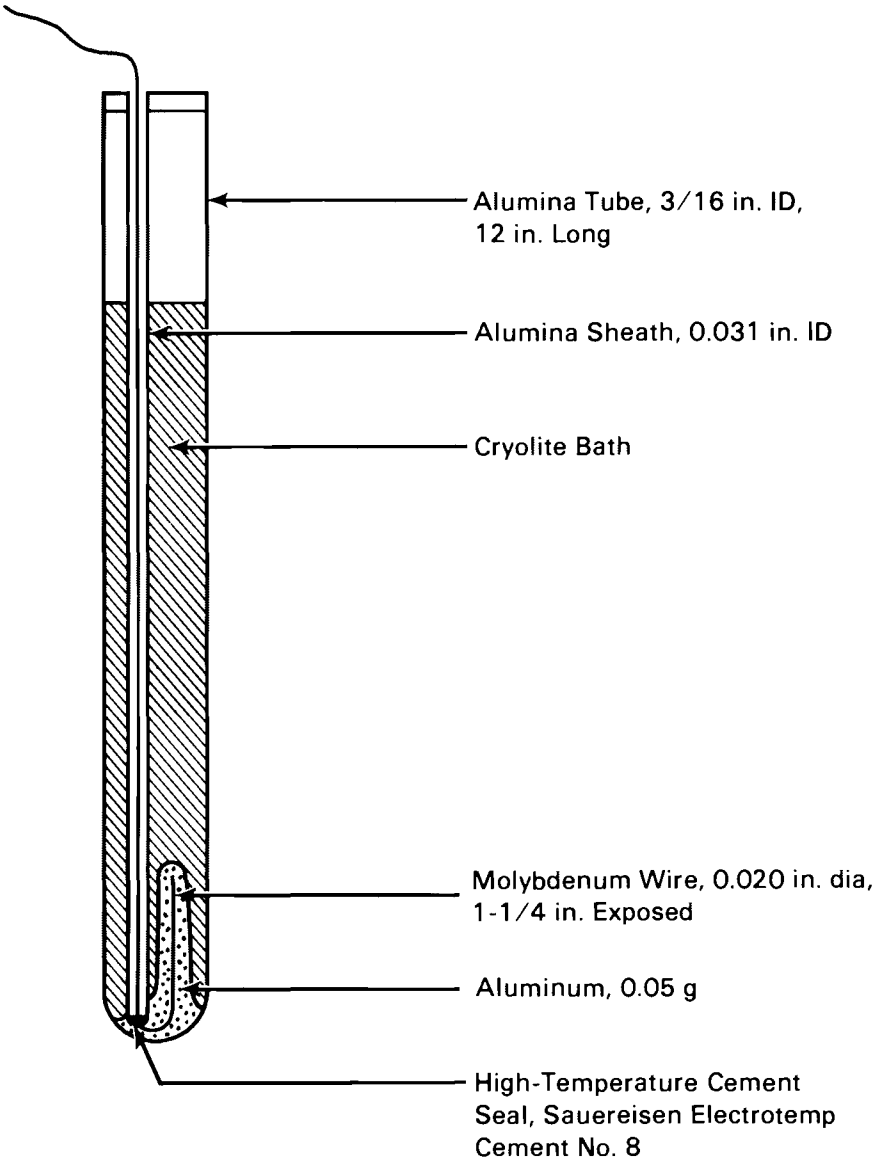

FIGURE 6. Reference Electrode (Burgman, Leistra, and Sides 1986)

ELECTROLYTE COMPOSITION

The electrolyte composition GM#2 was used in all of the cell development experiments. In addition, excess alumina powder was added to the cryolite bath to protect the various alumina-sheathed cell components from gradual dissolution. The amount of alumina added in each case was twice the weight required for saturation.

Post-test optical analysis of the frozen bath showed a fairly uniform electrolyte matrix of cryolite and alumina. The alumina did not appear to "settle out." Post-test inspection showed very little dissolution of the various alumina cell components over a 6-h test period.

TEMPERATURE CONTROL

Initial studies had problems associated with the furnace and its controller. The potentiostat was found to be sensitive to current/voltage fluctuations in the external devices. Electromagnetic interference (EMI) generated by the furnace resulted in widely varying current and voltage readings at the potentiostat, which caused the unit to overload. Various attempts were made to eliminate this problem, including the use of different furnaces and controllers and the incorporation of EMI shields around the electrode assemblies. These attempts were unsuccessful so data were collected in the earlier studies with the furnace turned off. The problems were later corrected by using an isolation transformer to eliminate high-level transients in the line cord. In addition, a furnace with noninductively wound elements was used to correct potential problems from continuous ac signal pickup.

In earlier studies, the temperature was allowed to climb to about 1040°C and then the furnace was shut off to prevent EMI. Data were collected with the furnace off over a period of about 10 min or until the temperature had fallen to about 970°C. With the indicated design improvements, studies are now being conducted under isothermal conditions.

PRE-ELECTROLYSIS

Pre-electrolysis of molten cryolite is sometimes performed to remove electroactive bath impurities. No pre-electrolysis was performed in this work, again due to problems associated with the furnace. The potentiostat was unable to control cell current and voltage when the furnace was on. However, a recent experiment performed using the new furnace indicated that pre-electrolysis may not be necessary (in most cases). Polarization data collected using a platinum anode shortly after its immersion in freshly prepared molten cryolite showed no residual currents below the theoretical decomposition potential for alumina. This finding suggests that no significant amounts of impurities (which would give rise to these currents) were present.

EXPERIMENT DURATION

In the cell development work, experiment duration varied widely depending on the number of variables examined in a given run. In future anode evaluations, the following steps and their approximate durations are expected:

Steps	Hours
thermal equilibration/melting	1.5
electrode insertion/submersion tests	0.2
pre-electrolysis (may not be required)	0 to 3.0
ac impedance study on control anode (optional)	0.5
dc polarization study on control anode	0.5
ac impedance study on test specimen	0.5
dc polarization study test specimen	0.5
optional ac impedance studies	0 to 1.0
	3.7 to 7.7

The optional ac impedance studies include experiments performed at dc potentials different than the open circuit potential. In addition, other types of polarization experiments may be conducted, including dc polarization as a function of time or environmental factors.

ATMOSPHERE CONTROL

Precautions were taken to minimize variations in vapor phase composition. In particular, the molten salt bath was continuously blanketed with argon gas to reduce the levels of water vapor and facilitate the removal of oxygen (generated during electrolysis) above the liquid.

Significant amounts of oxygen gas were detrimental only when graphite was present as a cell component. Since the cell designs chosen for the present work used no graphite, the presence of oxygen was considered neither harmful nor nonrepresentative of actual Hall-Heroult cell conditions. In most experiments, purging with argon gas was used merely to maintain a steady-state atmospheric composition.

CELL CONTAINER

Alumina was used as the cell container material for all of the experiments in this work. In a typical experiment, a 100-mL crucible was used. All of the electrodes and secondary probes were sheathed in alumina. To minimize dissolution of the crucible and sheathed components, the molten cryolite bath was saturated in alumina.

An interesting phenomenon observed in this work was the tendency of cryolite to appear on the outside of the alumina container after a test was terminated. SEM/EDX analysis was performed on a cross section of an alumina crucible (which had contained molten cryolite) to determine whether the container was at all permeable to the bath. No significant amounts of cryolite-related chemical species were detected in the alumina container walls, suggesting that the mode of transport of cryolite was surface diffusion and/or evaporation/condensation. To minimize problems associated with "moving" cryolite, the alumina crucible walls should be reasonably high or a second high-walled container should be used.

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