Electrochemical Reduction of Aluminum in Ionic Liquids

Donna L Baek, Bryna Joann Haile, Mary E Case, Tedd E Lister

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Idaho National Laboratory Idaho Falls, Idaho 83415

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Sub-Task: Electrochemical Reduction of Aluminum in Ionic Liquids Donna L. Baek¹, Bryna J. Haile², Mary E. Case¹, Tedd E. Lister¹ ¹Idaho National Laboratory, ²Idaho State University



INTRODUCTION

Electrorefining is an electrochemical process used to increase purity of a metal. The Hoopes process is the current state-of-the-art for purifying Al. This is an electrochemical process that uses three molten layers at temperatures of 700 °C to 900 °C with an energy consumption of 15-18 kWh/kg.^[1] An energy efficient alternative is using ionic liquids. A room temperature ionic liquid is a solvent made of ions that is liquid at temperatures below 100 °C. The advantages of using ionic liquids for electrorefining are low energy and electrode consumption, low pollutant emission, and low operating costs.^[1]

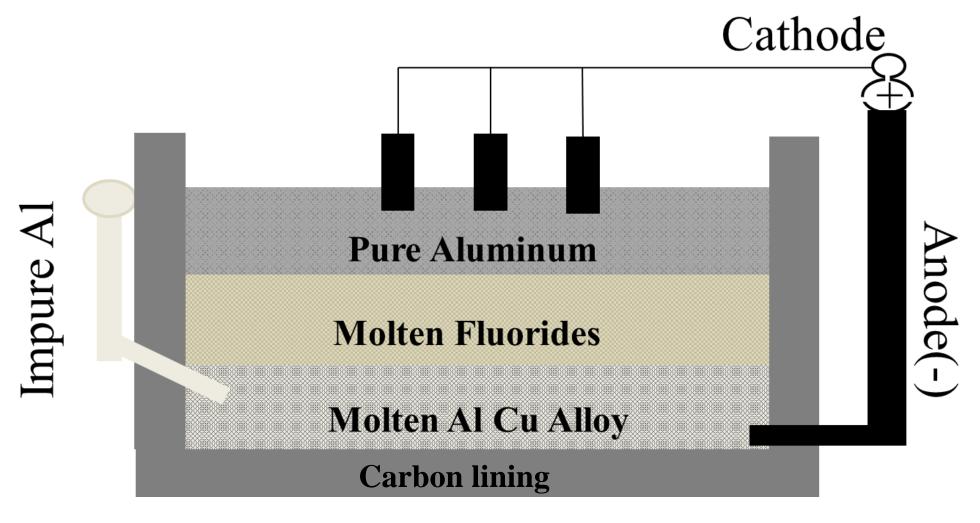


Figure 1: Diagram of Hoopes electrolytic cell

EXPERIMENTAL

Aluminum chloride (AlCl₃) was added to 1-Ethyl 3-methyl imidazolium chloride (EMIC) to create a 1.2:1 molar ratio solution (Figure 2). Experiments used a Bio-logic SAS potentiostat with a Cu working electrode, Al counter and reference electrode (Figure 3).

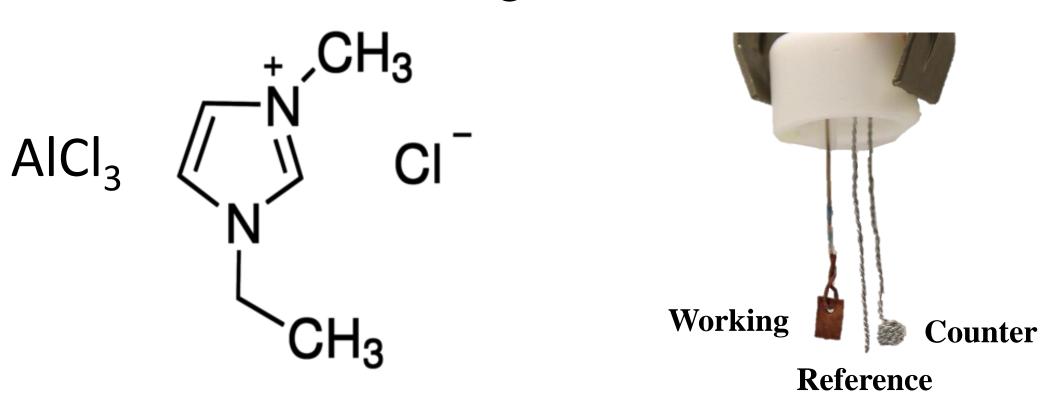


Figure 2:Chemical structure of 1.2 AlCl₃ 1-Ethyl 3-methyl imidazolium chloride.

Figure 3: Cu working, Al counter, and Al reference electrodes

Experiments were conducted at 80, 90, 100, 110 °C and potentials of -0.3, -0.4, -0.5 V. Current efficiency was determined by the following equation:

Current Efficiency =
$$\frac{Actual\ Mass}{Theoretical\ Mass} \times 100$$

The working electrode was rinsed with water and methanol, and then images were taken with the Scanning Electron Microscope (SEM).

RESULTS AND DISCUSSION

Cyclic Voltammograms (CV) taken of the 1.2:1 AlCl₃:EMIC (Figure 4) showed where Al oxidation and reduction occurred. The CV taken were used to determine what potential chronoamperometry (CA) experiments were performed. Electrodeposition of Al was successful, as shown in Figure 5.

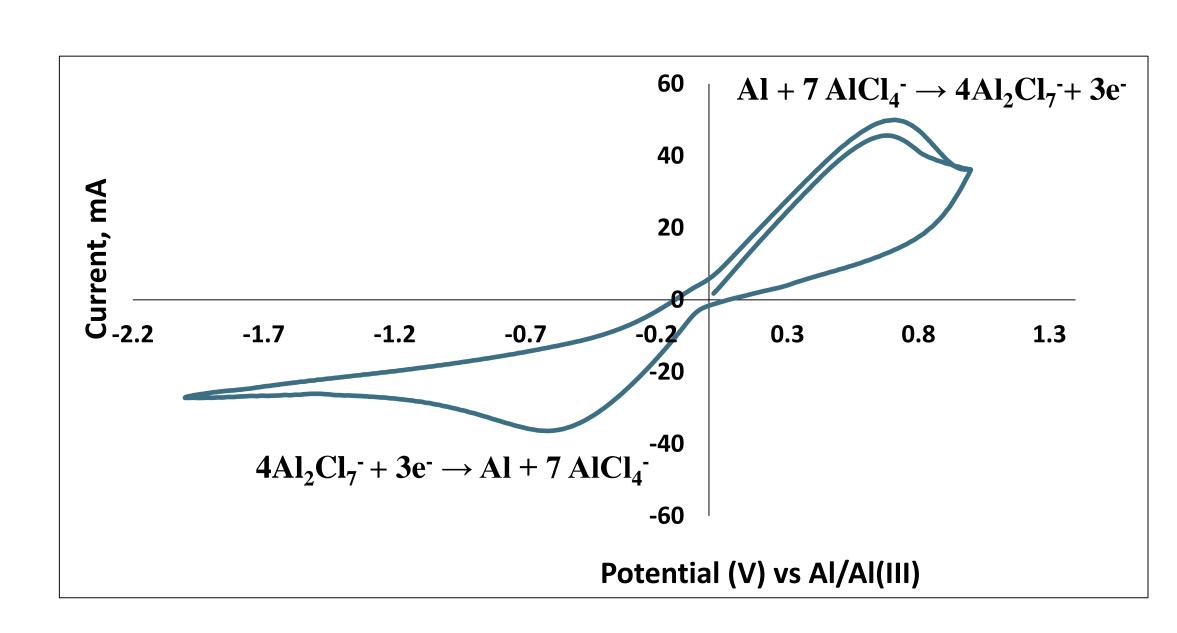


Figure 4: Cyclic voltammogram of Al redox in 1.2:1 AlCl₃:EMIC

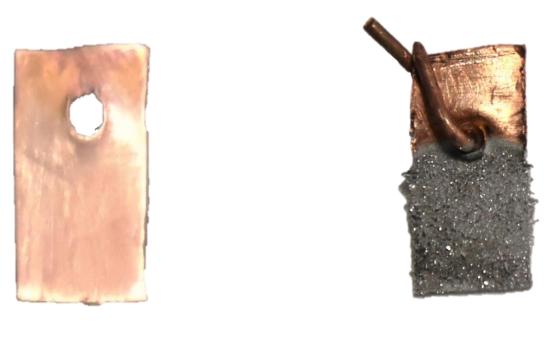


Figure 5: Cu working electrodes. Left: before deposition. Right: after Al deposition, showing dendritic growth.

It was observed that current efficiency is dependent on the potential and temperature of the solution (Figure 6). Dendrite growth occurred when a CA was conducted using a potential of -0.5 V. The SEM images (Figure 7) taken of working electrodes showed that at varying temperatures size and shape of aluminum particles change. As temperature increased, the particles became more jagged and larger. This indicates that temperature influences aluminum deposition.

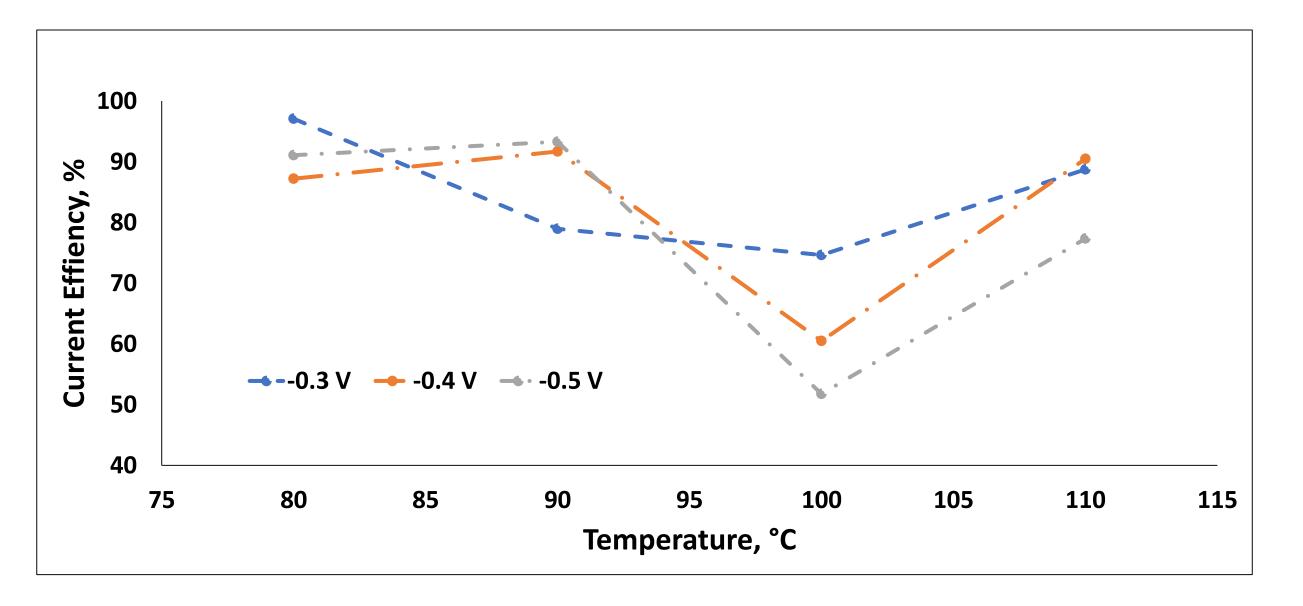
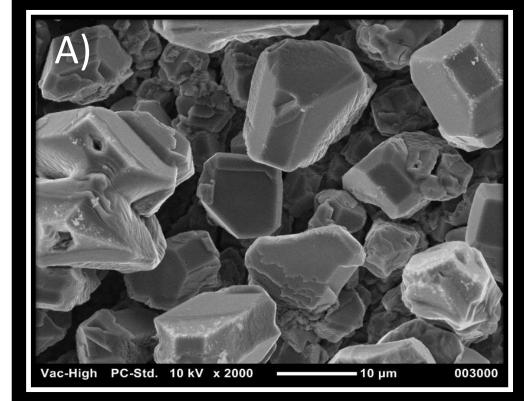
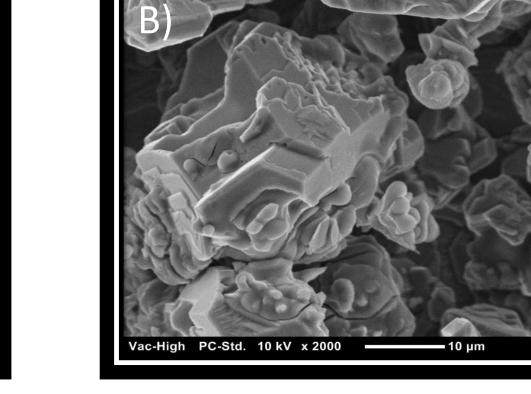


Figure 6: A graph showing the trend of current efficiency as a function of temperature.







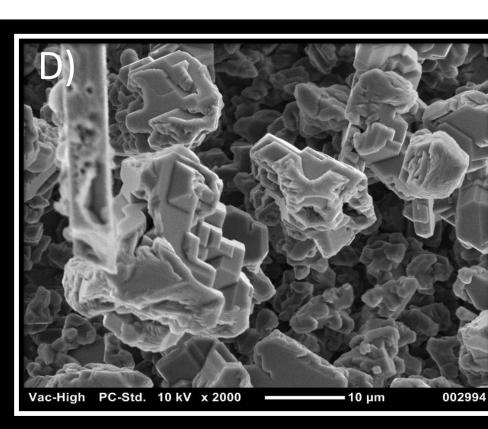


Figure 7: SEM images of aluminum plated onto the working electrode at -0.4 V. Taken with a zoom of 10kV x 2000. A) 80 °C, B) 90 °C, C) 100 °C, D) 110 °C

CONCLUSIONS

Deposition of aluminum onto the working electrode was achieved. The deposition is dependent on temperature and potential used while performing the CAs. When a CA was conducted at a potential of -0.5 V, particles appeared rougher. Thus, provided more nucleation points for dendritic growth. The experiments conducted indicated which potentials temperatures and are most advantageous for a dendrite free deposition. This process uses ionic liquids, which greatly reduces the hazards and energy requirements associated with current industrial processes.

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[1] Kondo, Mitsuhiro, Hideo Maeda, and Mikio Mizuguchi. "The production of high-purity aluminum in Japan." JOM 42.11 (1990): 36-37.

