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In Situ X-ray Diffraction Investigation of the Evolution of Pb-Oxide/Pb-Sulfate Surface Layers on Pb-Alloy Anodes

Wednesday, 25 November 2015 14:00 (30 minutes)

This presentation will describe the quantitative measurement, by in situ X-ray diffraction (XRD) and subsequent Rietveld-based quantitative phase analysis and thickness calculations, of the evolution of the lead dioxide and lead sulfate surface layers formed on a number of lead alloy anodes under simulated copper electrowinning conditions. A novel electrochemical flow cell is also described. The work is the first truly in situ XRD study of the surface layer evolution on lead alloy substrates under cycles of galvanostatic (electrowinning) and potentiodynamic (power interruption) conditions, and as such is of key interest to the metallurgical and lead acid battery communities. In a general sense, the in situ results show that the polymorph of lead dioxide forms immediately on the anode under galvanostatic conditions, and undergoes continued growth until power interruption where it transforms to lead sulfate. The amount of residual lead dioxide increases with the number of cycles due to incomplete conversion to lead sulfate, which affects the electrochemical performance of the alloy. Specific variations in surface layer mineralogy and thickness as a function of cycle number and time are used to explain differences in electrochemical performance across the alloy suite.

Keywords

Electrochemical cycling, surface layer evolution, quantitative phase analysis

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