

DETECTION AND QUANTIFICATION OF PASSIVATION LAYERS IN ELECTROCHEMICAL INERT ANODES BY *IN SITU* AND *EX SITU* DIFFRACTION

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Currently, many important light metals, including aluminium, magnesium and titanium, are produced via electrowinning involving the use of carbon anodes in the reduction of solid metal oxides in a molten salt bath (for example, cryolite, Na_3AlF_6 or calcium chloride, CaCl_2) operating at $\sim 1000^\circ\text{C}$. The carbon anodes can contaminate the light metal product and are consumed at a rapid rate, producing carbon dioxide emissions, necessitating frequent replacement, all of which affects cell stability and reduces cell efficiency.

Inert anodes are a potential replacement for carbon anodes as they are not consumed and evolve only pure oxygen from the melt. As well as reducing costs and greenhouse gas emissions, the inert anode provides greater cell stability and avoids carbon contamination of the product.

Preliminary *ex situ* studies suggest that passivating layers form on the inert anode surface upon immersion in the molten bath and wear away during cell operation. These layers are thought to be the key to electrode longevity as they protect the anode from the molten bath. Fundamental study of these layers *in situ* in operating cells is imperative in understanding the mechanism of their formation and wear, and hence, to developing genuinely breakthrough inert anode technology.

An *in situ* tomographic study of cold electrochemical cells to observe passivation layers formed on inert anodes was conducted using energy-dispersive synchrotron X-ray diffraction at Station 16.4 at the Daresbury Synchrotron Radiation Source. That work found that the energy-dispersive technique was able to be applied to the quantification of passivation layers. The findings of the previous *in situ* study are confirmed by an *ex situ* microscopy and diffraction study on anodes that were removed from the same electrochemical cells. The energy-dispersive analysis technique has also been applied to an operational electrochemical cell using the JEEP beamline at the Diamond Light Source.

An overview of the research project and the findings of the *ex situ* confirmatory study will be presented. Initial results from the *in situ* operational cell diffraction studies will also be given.