

Operando analysis of lead cells using neutron scattering



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Abstract

Lead cells are made of very heavy materials that do not allow deep penetration of X-ray probes. In the positive electrode the charge/discharge conversion process is PbO_2 PbSO_4 , while in the negative electrode is Pb PbSO_4 . Consequently, the only way to obtain information on the phase evolution within thick commercial lead cells is to use neutron probes, due to their high penetration in high electron density materials. We will review the use of two different neutron scattering techniques: gauge-volume neutron diffraction and wavelength-selective neutron imaging by Bragg-edge analysis. For gauge-volume neutron diffraction, we used a neutron diffractometer equipped with a set of collimators and controlled slits, allowing operando studies with both spatial and time resolution. The experiments described were performed in a time-of-flight instrument (VULCAN) at the Spallation Neutron Source, Oak Ridge National Laboratory (Tennessee, USA). Using the wavelength-selective neutron imaging technique by Bragg-edge analysis, we compared the transmission images just before and after a Bragg-edge. We will show that the concentration of the crystallographic species can be related to this ratio of transmission images. The experiments discussed were mainly performed in the ICON instrument at the Paul Scherrer Institut (Switzerland).

Bio

Ángel Larrea obtained his PhD in Nuclear and Particle Physics (1991), working on new X-ray detectors using superconducting materials. He combined Monte Carlo simulations with magnetic measurements at very low temperatures (20 mK). After his postdoc at the CNRS (Toulouse, France), he joined the CSIC (ICMA, Zaragoza, Spain). There he worked mainly on the microstructure of ceramic materials. He launched and directed (2000–2012) the Electron Microscopy Service of the University of Zaragoza. He has worked on high temperature superconductors, directionally solidified ceramic eutectics, ancient materials (lustre ceramics), and solid oxide cells (fuel cells and electrolyzers), trying to understand the relationships between the microstructure and the physical properties of the materials. In recent years, he has led several industrial projects for the development

of new lead battery technologies. In particular, a project with the multinational company Exide and NIST (USA) to apply neutron scattering techniques to operando studies. He has been a member of the Commission of the National Evaluation and Foresight Agency of the Spanish Government (2010-2013) and Chairman of the Evaluation Panel of the Spanish State Research Agency (AEI) for the strategic line A new generation of batteries (2021).