

Received 16 April 2015
Accepted 16 April 2015

Keywords: PbO_2 ; PbSO_4 ; electrochemical cycling; flow cell; *in situ* X-ray diffraction; Rietveld refinement; quantitative phase analysis.

In *situ* synchrotron X-ray diffraction investigation of the evolution of a $\text{PbO}_2/\text{PbSO}_4$ surface layer on a copper electrowinning Pb anode in a novel electrochemical flow cell. Corrigendum

Marie Clancy,^{a*} Mark J. Styles,^b Colleen J. Bettles,^a Nick Birbilis,^a Miao Chen,^c Yansheng Zhang,^c Qinfen Gu,^d Justin A. Kimpton^d and Nathan A. S. Webster^{c*}

^aARC Centre of Excellence for Design in Light Metals, Department of Materials Engineering, Monash University, Clayton, VIC 3800, Australia, ^bCSIRO Manufacturing Flagship, Bayview Avenue, Clayton, VIC 3168, Australia, ^cCSIRO Mineral Resources Flagship, Private Bag 10, Clayton South, VIC 3169, Australia, and ^dAustralian Synchrotron, 800 Blackburn Road, Clayton, VIC 3168, Australia. *Correspondence e-mail: marie.clancy@monash.edu, nathan.webster@csiro.au

Figures 7 and 8 of the article by Clancy *et al.* [(2015), *J. Synchrotron Rad.* **22**, 366–375] are corrected.

In the article by Clancy *et al.* (2015), Fig. 8(a), showing the results of quantitative phase analysis, was incorrect. The values shown in the published manuscript were determined using a preliminary and incorrect model in the Rietveld

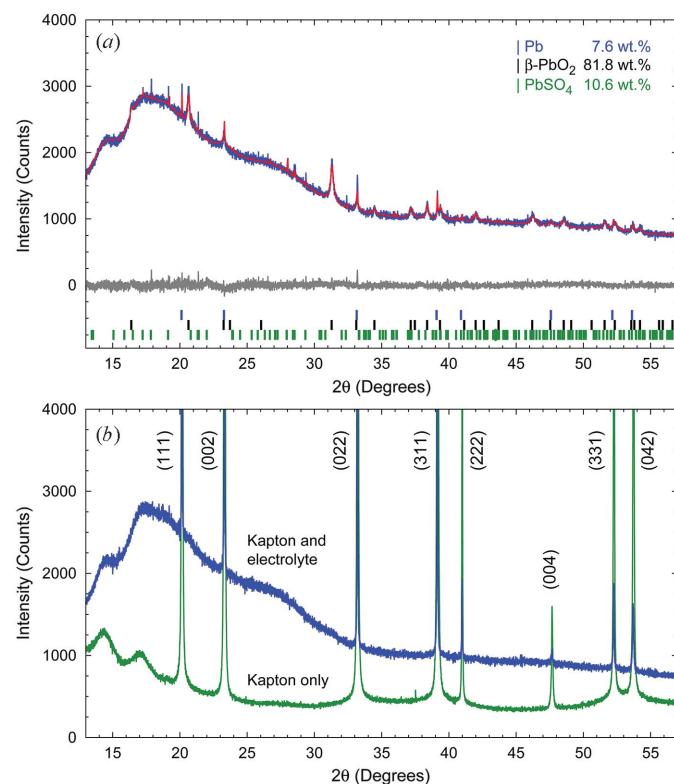
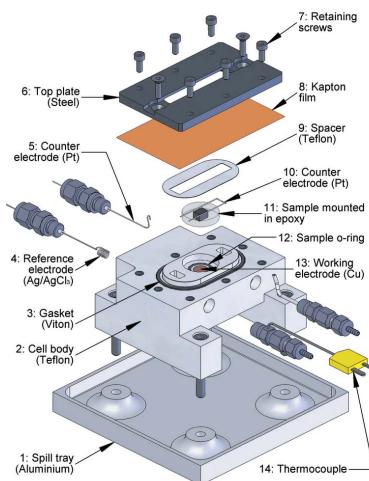
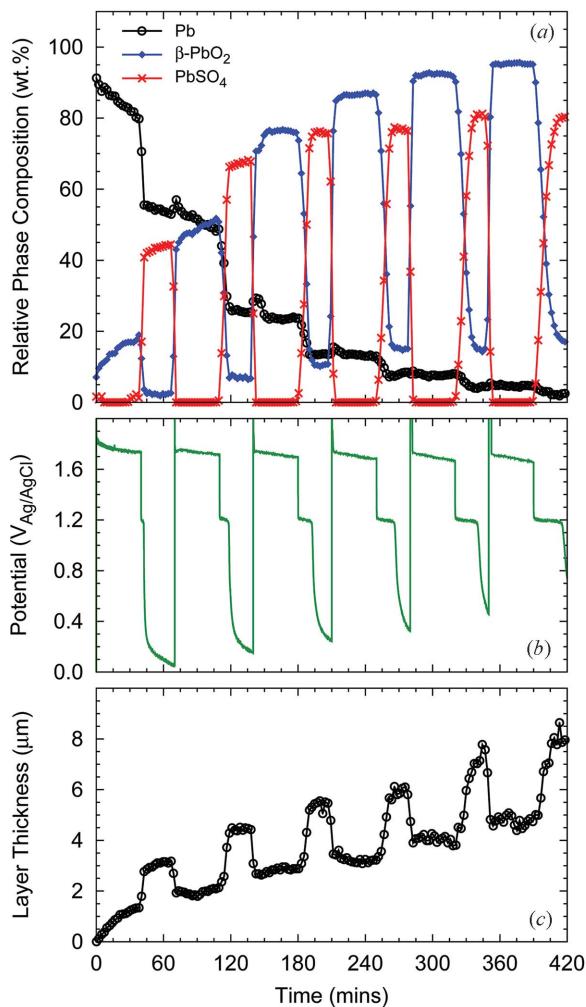


Figure 7
(a) Rietveld refinement output of a dataset collected during the early stages of the OCP segment of the fifth cycle ($t = 320$ min, $R_{wp} = 2.56$). The experimental data are shown as a blue solid line, the calculated pattern as the red solid line, and the difference pattern as the grey solid line below. The tick marks below the difference curve are the Bragg reflection markers for Pb (upper), β - PbO_2 (middle) and PbSO_4 (lower). (b) Overlay of datasets collected at the beginning of the first GALV segment (upper), and for the substrate and Kapton® film before the flow of electrolyte commenced (lower). The Pb reflections are labelled with their Miller indices.

**Figure 8**

(a) Results of Rietveld refinement-based quantitative phase analysis, showing the evolution in relative concentration of the Pb substrate and the PbO₂ and PbSO₄ surface layers during the electrochemical test. (b) Potential *versus* time plot. (c) Estimated PbO₂/PbSO₄ surface layer thickness as a function of time.

refinements. The correct Fig. 8, determined using the correct model, is shown here. Due to this error, the phase concentration values quoted in Fig. 7(a) are incorrect; the correct Fig. 7 is also shown here.

Owing to error in Fig. 8(a) there are two values which are incorrect in the text immediately below the figure on page 373 of the original article. The first sentence below the figure should read ‘PbO₂ formed immediately on the substrate, and continued to grow during the GALV segment of the first cycle, as indicated by the increase in crystalline phase concentration from 7 wt% at $t = 0$ min to 19 wt% at the end of the segment ($t = 38$ min).’ In addition, the second sentence of the third paragraph on page 374 should read ‘Since PbO₂ was present on the surface of the anode at $t = 0$ min [the concentration of PbO₂ in Fig. 8(a) is 7 wt%], the I_0 value used here in equation (6) is not the true I_0 value for a layer-free surface, which adds to the semi-quantitative nature of this approach.’

The errors occurred during the manuscript revision process, and correction of these errors does not change the outcomes or interpretations of the article.

References

- Clancy, M., Styles, M. J., Bettles, C. J., Birbilis, N., Chen, M., Zhang, Y., Gu, Q., Kimpton, J. A. & Webster, N. A. S. (2015). *J. Synchrotron Rad.* **22**, 366–375.