

# Post-Mortem Analysis of Calendar-Aged 16 Ah NMC/Graphite Pouch Cells for EV Application

Amaia Iturrondobeitia,<sup>†,‡</sup> Frederic Aguesse,<sup>\*,†,§</sup> Sylvie Genies,<sup>‡,§</sup> Thomas Waldmann,<sup>||,¶</sup> Michael Kasper,<sup>||</sup> Niloofar Ghanbari,<sup>||</sup> Margret Wohlfahrt-Mehrens,<sup>||</sup> and Emilie Bekaert<sup>†</sup>

<sup>†</sup>CIC energiGUNE, Albert Einstein 48, 01510 Miñano, Alava, Spain

<sup>‡</sup>Université de Grenoble Alpes, F-38000 Grenoble, France

<sup>§</sup>CEA, LITEN, F-38054 Grenoble, France

<sup>||</sup>ZSW – Zentrum für Sonnenenergie- und Wasserstoff-Forschung Baden-Württemberg, Helmholtzstrasse 8, D-89081 Ulm, Germany

## Supporting Information

**ABSTRACT:** Application of Li-ion batteries for transportation not only requires long cycling life but also the preservation of the electrochemical performance during the resting period. For certain car usage this resting time could be predominant compared with the cycling activity and is referred to as calendar aging. To understand the aging mechanisms during calendar aging, an extensive post-mortem study was conducted on commercial 16 Ah NMC/graphite pouch cells stored at 5, 25, 45, and 60 °C. The post-mortem analyses were performed in parallel within three separate laboratories across Europe. They included visual inspection and structural and microstructural analysis along with a combination of analytical techniques to determine accurately the composition of positive (NMC) and negative (graphite) electrodes and the electrolyte.

A direct correlation was established between the calendar-aging temperature and the degradation of the cells. The measurements revealed a severe deterioration phenomenon for the electrodes aged at 45 and 60 °C. These results are explained by the formation of a resistive interface on top of the negative electrodes due to a continuous and heterogeneous growth of a surface layer. Electrochemical impedance spectroscopy and electrochemical measurements confirm the resistance increase during cell degradation. At high temperatures, this occasionally leads to a Li deposition phenomenon. Nonetheless, we revealed that this degradation process does not affect the bulk structure of the materials but only the surface of the particles.



## ACKNOWLEDGMENTS

The research leading to these results has been performed within the MAT4BAT project (<http://www.mat4bat.eu>) and received funding from the European Community's Seventh Framework Program (FP7/2007-2013) under grant agreement no. 608931. Special thanks are addressed to our partners who have carried out the calendar tests (Figure 1) from which the cells studied in this paper have been collected, namely: CEA-INES (Dr. Arnaud Delaille and Bramy Pilipili Matadi), EIGSI (Dr. Francois Duclaud and Aurélien Guignard), CIDETEC (Dr. César Gutiérrez Couceiro), and VITO (Dr. Khiem Trad). We thank G. Arnold (ZSW) for conducting the ICP-OES measurements and C. Pfeifer (ZSW) for SEM analysis, C. Chabrol (CEA) for XRD measurements, J.-F. Martin (CEA) for electrolyte analysis, and I. Jimenez-Gordon (CEA) and L. Daniel (CEA) for helpful discussions.