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| ESRF | **Experiment title:**<br>**In-situ investigation of $AlCl_4^-$ anion intercalation in aluminum/graphite battery by SAXS** | **Experiment number**:<br>CH-5269 |
| **Beamline**:<br>BM26B | **Date of experiment**:<br>from: 15/11/2017 to: 20/11/2017 | **Date of report**:<br>05/10/2020 |
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**Report:**

The aluminum/graphite battery involving the $AlCl_4^-$ anion intercalation in the graphite can represent a new powerful and low-cost energy storage system [1,2]. A detailed investigation on such system can allow a deeper comprehension enabling a further optimization and the obtainment of high performances batteries. Thanks to the set-up available at BM26 beamline, Small Angle Scattering can be performed on the graphite cathode during the electrochemical process. This can allow us to obtain information on the micrometric and the structural reorganization changes of the electrode during the electrochemical process, obtaining a deeper understanding of a battery operation.

**The aim of the experiment is to investigate electrochemical process operando which involves the $AlCl_4^-$ anion intercalation in the graphite planes.** Accordingly, upon the electrochemical process of intercalation a variation of the graphite lattice planes distances was observed.

The reaction mechanism involving the intercalation of the $AlCl_4^-$ in the graphite was fully characterized by means of SAXS/WAXS. Graphite is a redox-amphoteric intercalation host and therefore able to yield so-called donor-type and acceptor-type Graphite Intercalation Compounds (GICs) via electrochemical intercalation of cations and anions at different potentials. The result of an intercalation reaction into graphite is a compound, the GIC, whose structure is characterized by a layered motif parallel to the graphene layers and a mostly ill-defined relationship between the graphite hexagonal network and the in-plane structure of the intercalant [3].

A total of four batteries test-cells (modified ad-hoc) were assembled and The sample holder was specially designed (Fig. 1). Two different samples represented by natural graphite and pyrolitc gaphite were investigated. The tes-cell were cycled very slowly (24/48 hours for one complete cycle) in order to obtain a comprehensive and detailed overview of the process. The measurements were performed at 12 keV evry 10 minutes. In figure two the SAXS/WAXS and the voltage profile collected simultaneosly upon cycling.

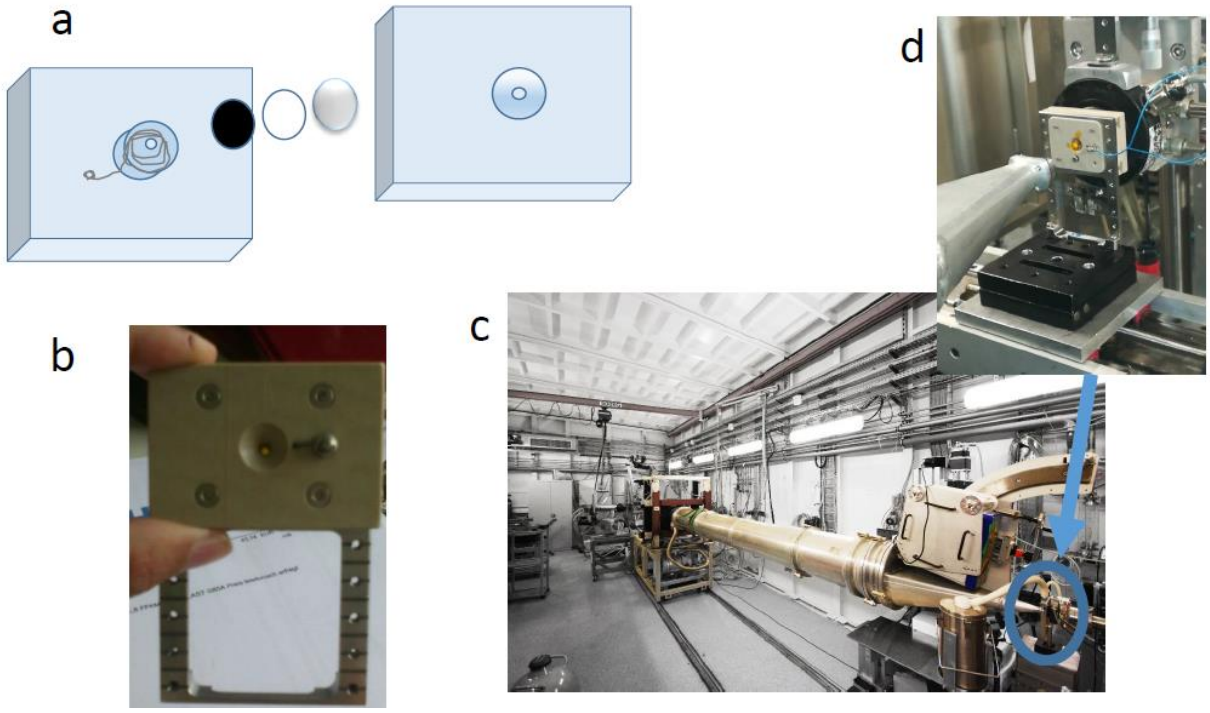


*Figure 1* A schematic view of the operando battery test-cell (a) and its picture (b). The BM26B set-up (c) and (d) and enlargement of the monted operando-cell are shown.

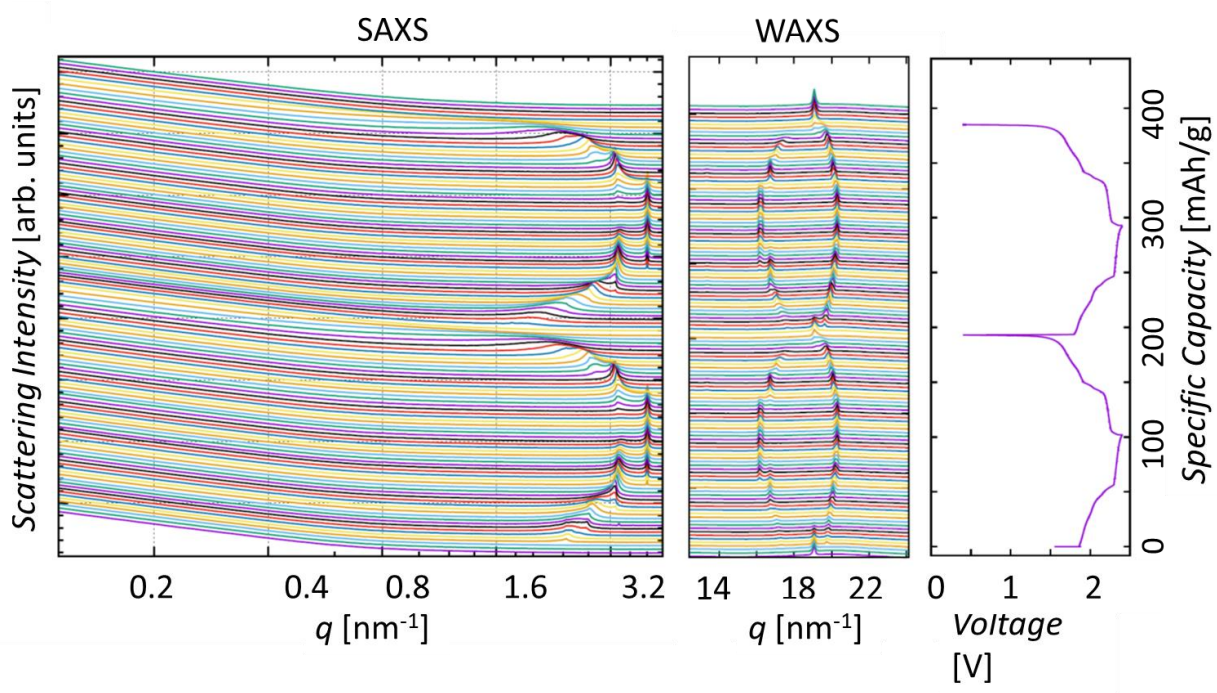


*Figure 2* SAXS and WAXS profiles collected upon the battery cycling compared with the voltage profile. The pattern were shifted for a better view of the process.


[1] G. A. Elia, K. Marquardt, K. Hoeppner, S. Fantini, R. Lin, E. Knipping, W. Peters, J.-F. Drillet, S. Passerini, R. Hahn, *Adv. Mater.* **2016**, *28*, 7564.

[2] M.-C. Lin, M. Gong, B. Lu, Y. Wu, D.-Y. Wang, M. Guan, M. Angell, C. Chen, J. Yang, B.-J. Hwang, H. Dai, *Nature* **2015**, *520*, 324.

[3] S. Rothermel, P. Meister, G. Schmuelling, O. Fromm, H.-W. Meyer, S. Nowak, M. Winter, T. Placke, *Energy Environ. Sci.* 2014, *7*, 3412.