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Insights on the electrochemical magnesiation of InSb from combined *operando* X-Ray diffraction and X-Ray Absorption Spectroscopy

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The continued acceleration of the lithium demand combined with its relatively low abundance and uneven concentration on the Earth's crust might dramatically increase its price in a near future. Mg-batteries are promising candidates to replace Li-ion batteries thanks to Mg abundance, theoretical capacity (2.2Ah/g - 3.8Ah/cm³), low cost and safety. However, metallic Mg reacts with standard electrolytes to form a blocking layer on its surface, preventing cation exchange, and thus dramatically limiting reversible stripping/deposition. An interesting alternative is to substitute Mg metal with another negative electrode made of *p*-block elements as they electrochemically alloy with Mg and possess adequate stability in standard electrolytes [1].

In a recent work, we investigated the electrochemical reactivity and performance of the InSb alloy as a negative electrode for Mg-ion batteries [2]. A strong synergy between In and Sb has been evidenced with the promotion of the electrochemical activity of Sb towards magnesiation along few cycles, in contrast to what was already reported in other studies.

Using complementary information from *operando* X-Ray diffraction (XRD) and X-Ray Absorption Spectroscopy (XAS), we further characterized in-depth the peculiar electrochemical behavior of InSb. *Operando* XRD measurements demonstrate the formation of the Mg₃Sb₂ phase almost all along the first magnesiation, accompanied by the extrusion of In metal. While crystalline MgIn has always been detected in the case of pure In or InBi electrodes [3], we observed a kinetically dependent electrochemically-driven amorphization of MgIn. This behavior suggests a possible competition between crystallization and amorphization in the material. EXAFS data obtained at the In and Sb *K*-edges at the ROCK beamline of synchrotron Soleil, corroborate the formation of Mg₃Sb₂ and In crystalline phases and the formation of MgIn, and give further insights on the atomic environment of In and Sb during the first magnesiation. Thanks to the evolution of EXAFS spectra as well as the changes in scattering paths, the phase proportions evolution as function of the number of Mg inserted into InSb has been followed.

These results are of paramount importance to shed light on synergetic effects between *p*-block elements and to study the effect of in situ amorphization on the electrochemical behavior.

References:

[1] Murgia *et al.*, *Electrochem. Commun.* **60**, 56 (2015)

[2] Blondeau *et al.*, *Phys. Chem. C* **123**, 1120–1126 (2019)

[3] Murgia *et al.*, *Electrochem. Acta.* **209**, 730-736 (2016)