



Large-scale exploration of the LiSiPON system using a high throughput experimental method

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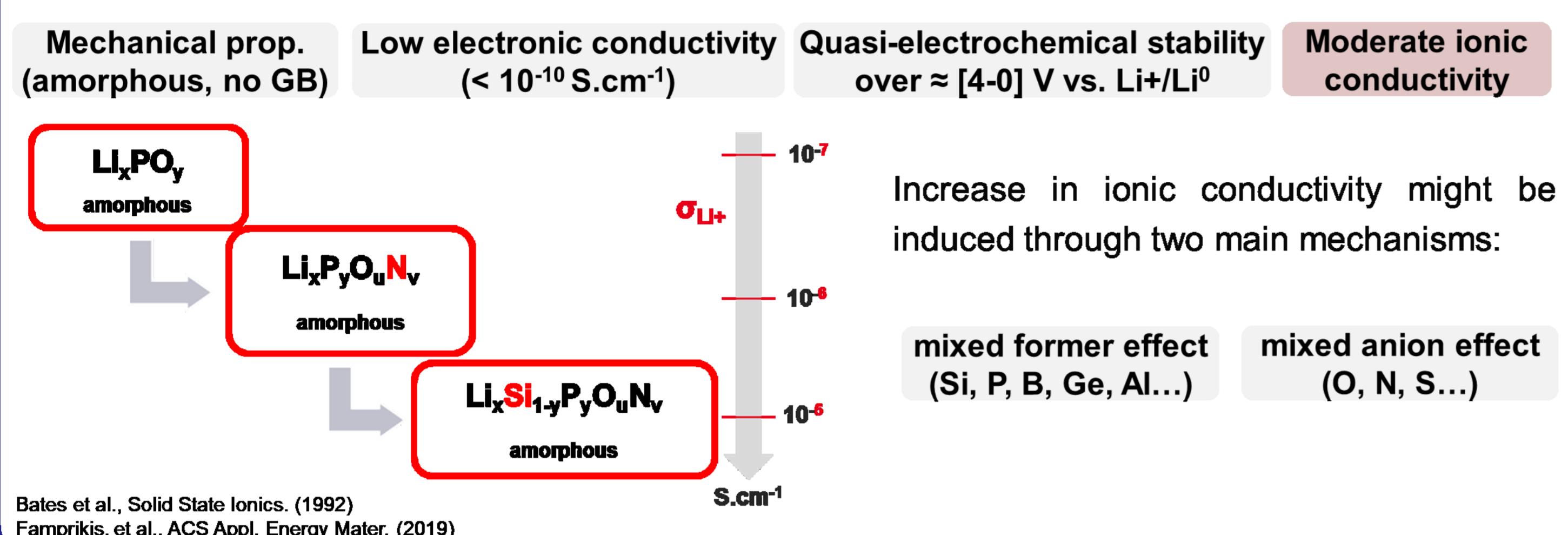
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Large-scale exploration of the LiSiPON system using a High-Throughput Experimental approach

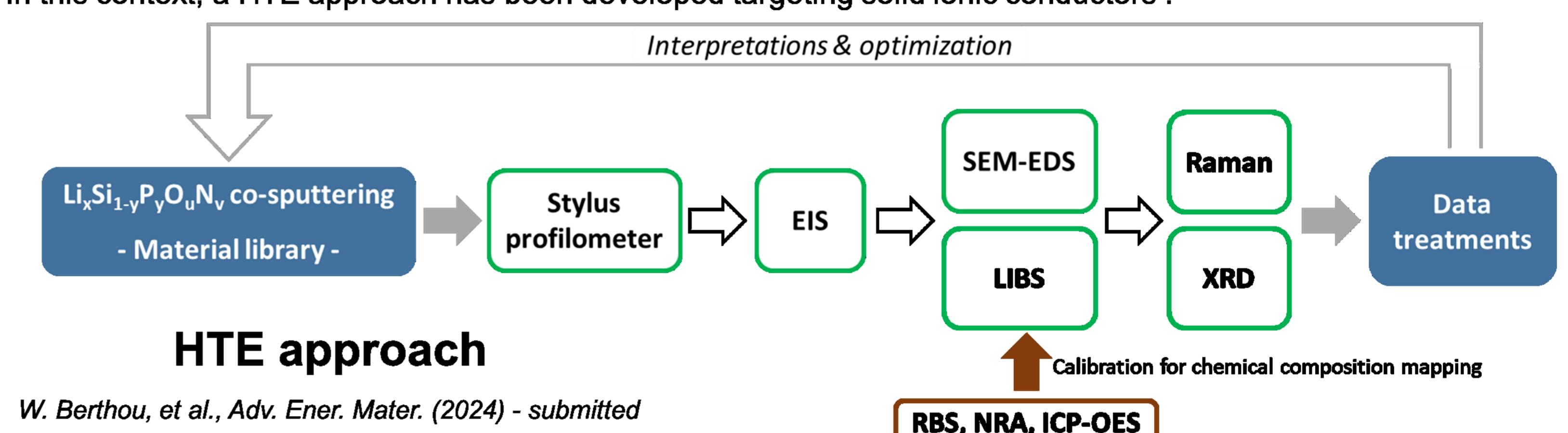
1. Introduction to HTE approach

Amorphous lithium phosphates are used since 25 years as state-of-the-art solid electrolyte into all-solid-state thin film micro-batteries. Those materials are worth of interest for their use as artificial SEI-CEI into All-Solid-State Lithium Batteries (Li-ASSB) :

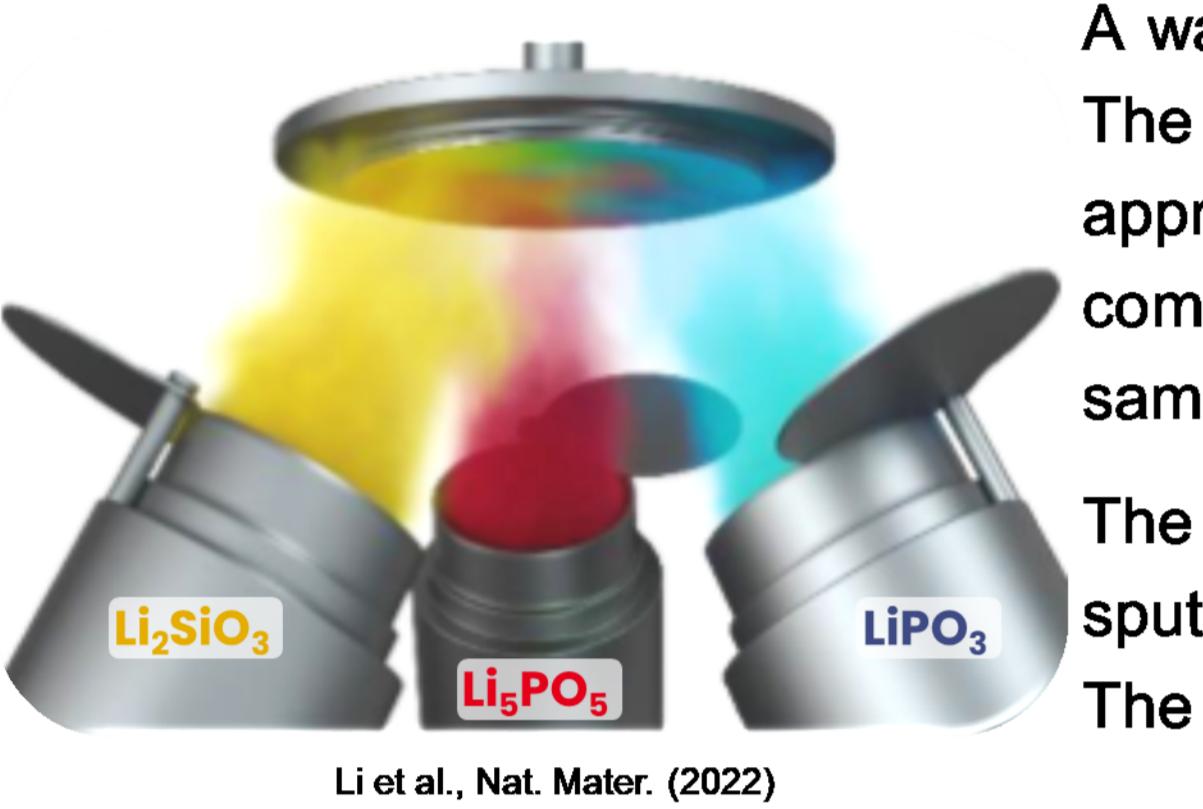


This is a place for a new paradigm, beyond 'try & errors' methods. High Throughput Experimentation (HTE) aims at coupling combinatorial methods (synthesizing wide material libraries) and automated, fast characterization techniques. In material science, it might accelerate the discovery of breakthrough materials by the study of complex systems (ternary, quaternary...).

In this context, a HTE approach has been developed targeting solid ionic conductors :

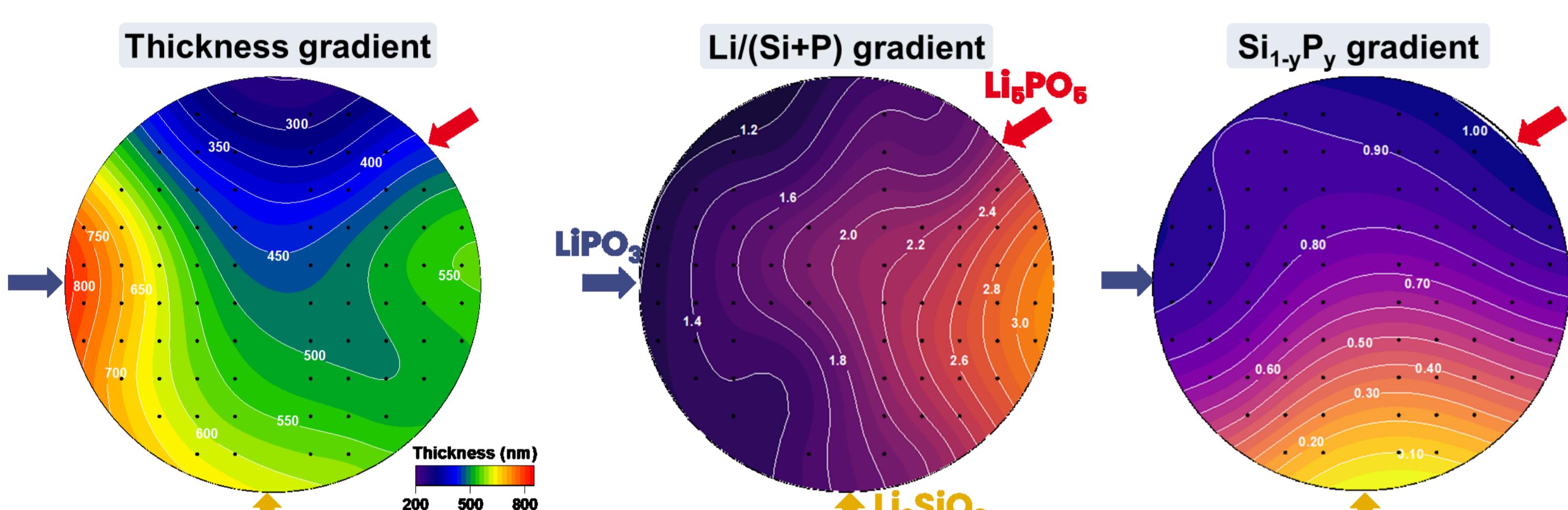


2. Combinatorial synthesis of thin films

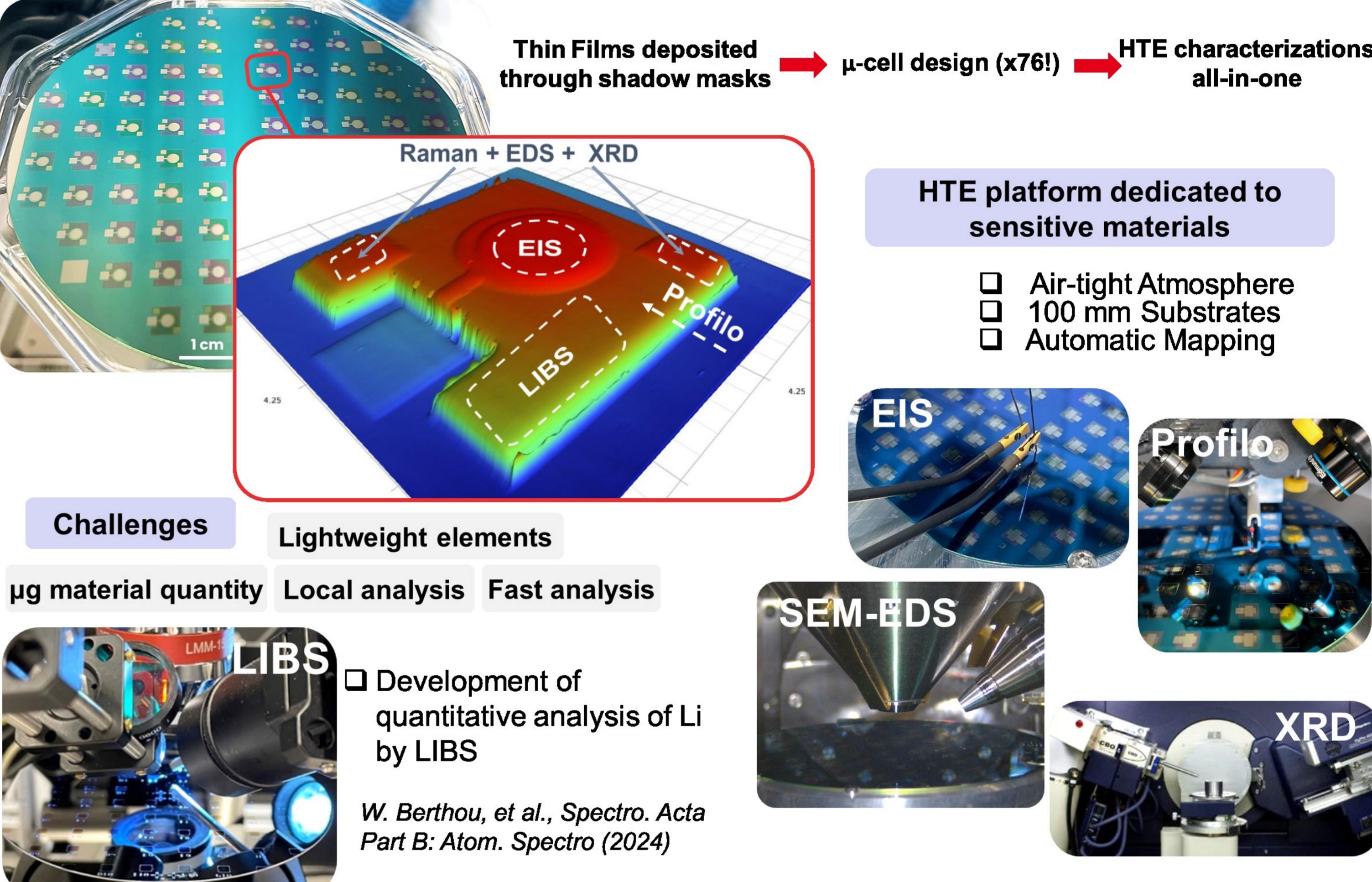


A way to achieve combinatorial synthesis is through co-sputtering. The PVD process owns interesting advantages i) simple/robust approach compared to other CS methods, ii) continuous composition & thickness gradients and iii) homogeneous batch of sample regarding synthesis parameters.

The $\text{Li}_{x}\text{Si}_{1-y}\text{P}_y\text{O}_{u,v}$ library is prepared on a 4" substrate by the co-sputtering of LiPO_3 , Li_5PO_4 and Li_2SiO_3 under pure N_2 atmosphere. The material library comprises thickness and composition gradients.

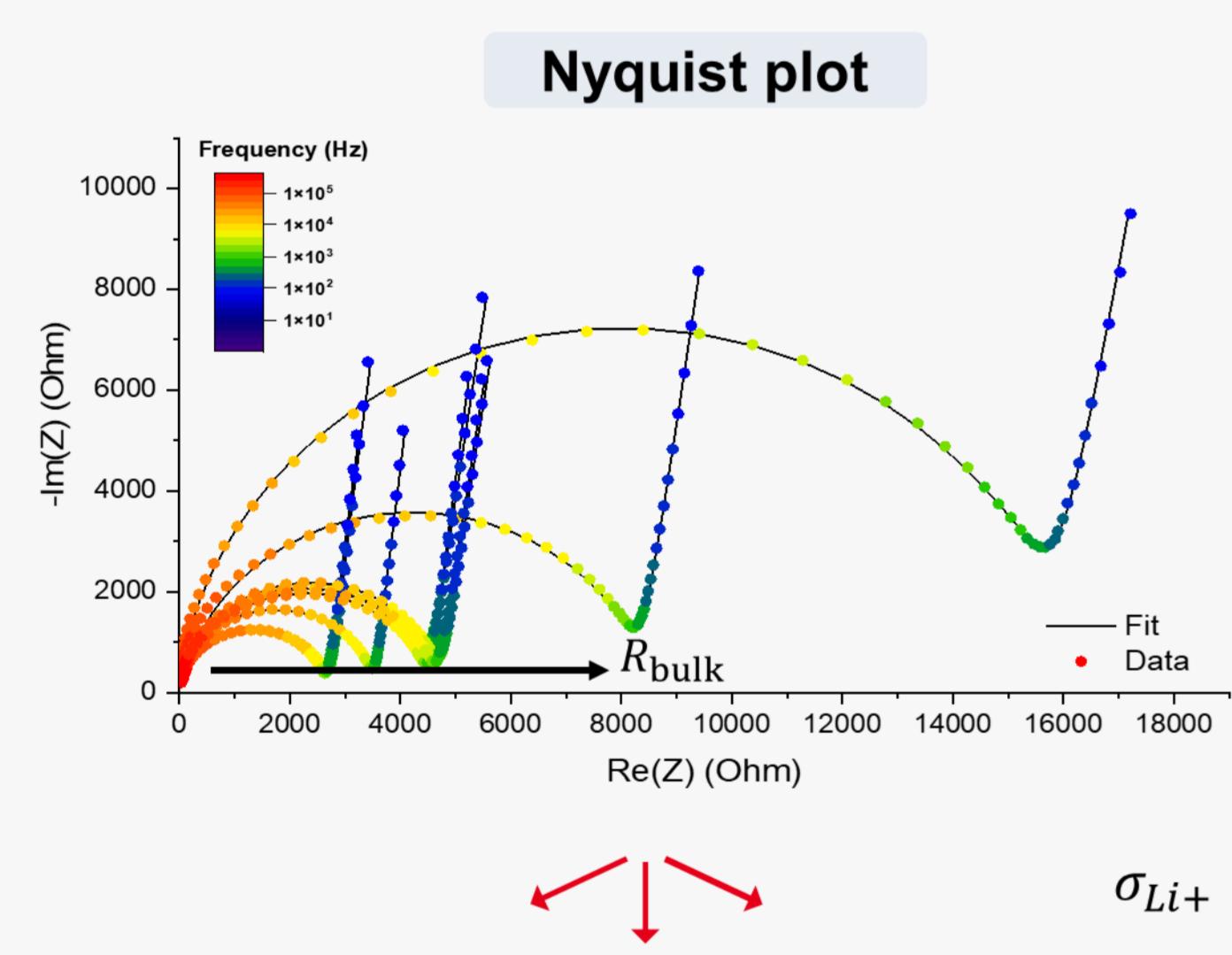


3. High-throughput characterizations

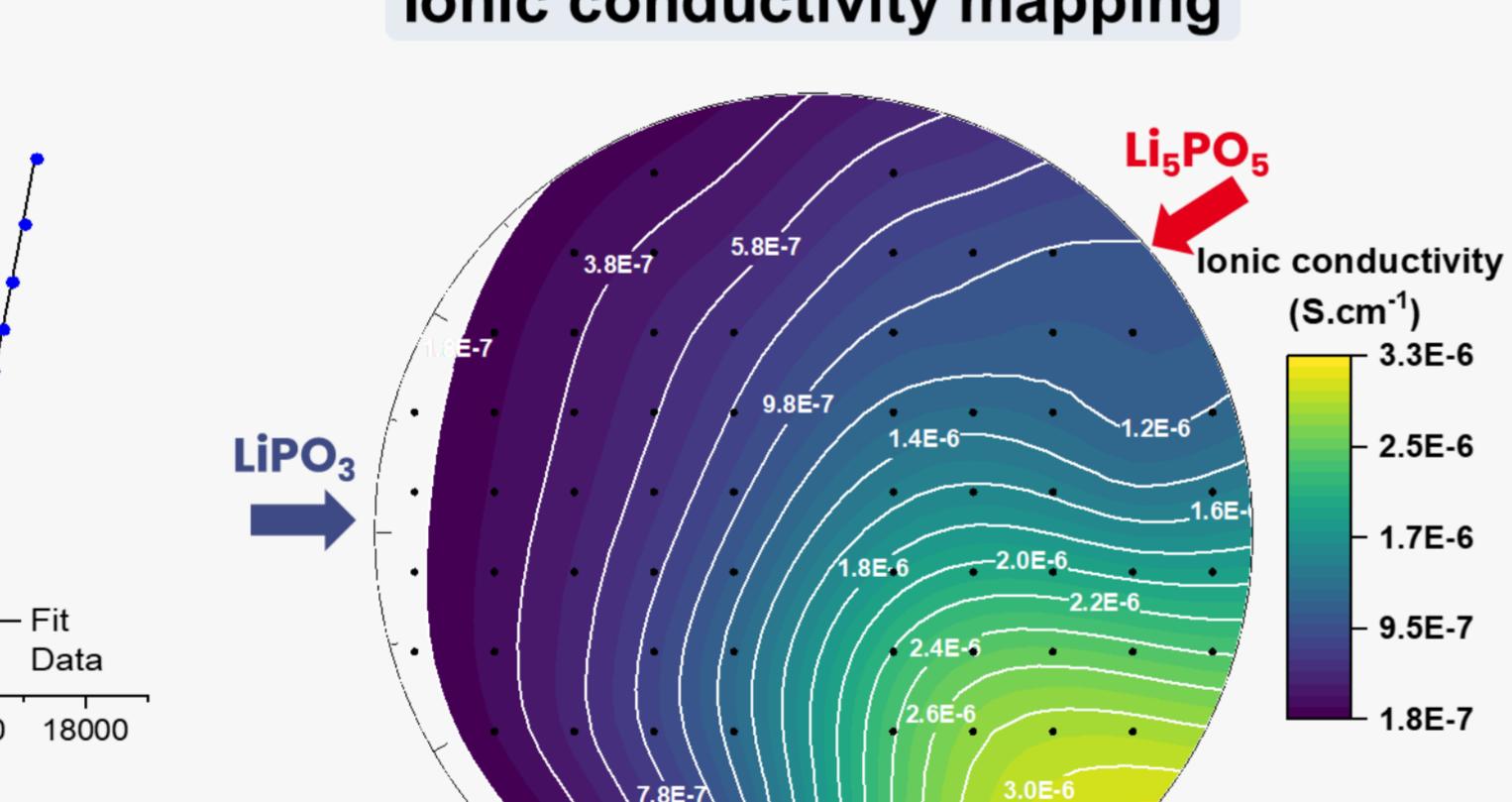


4. Correlative study of composition-structure-functional properties

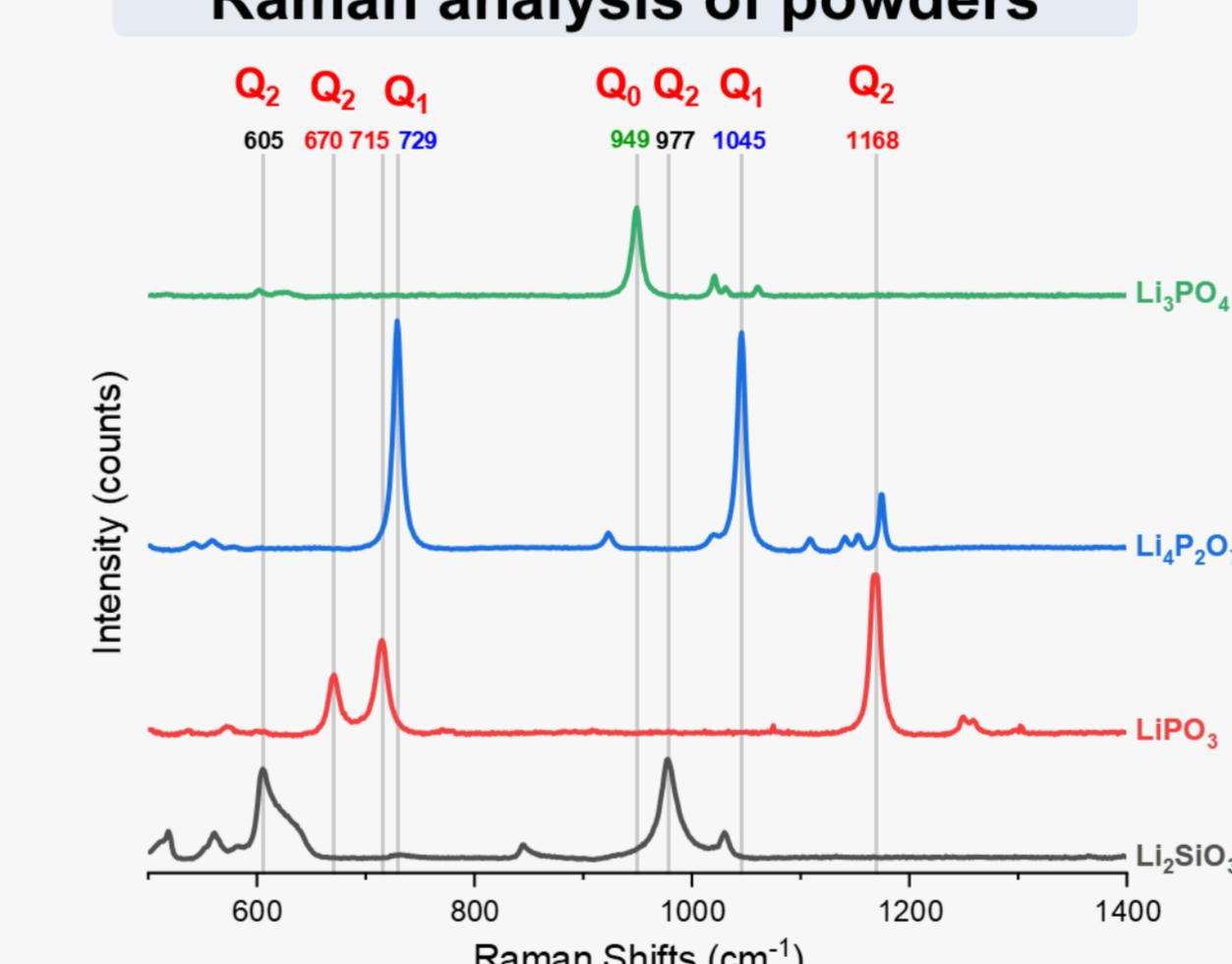
Functional properties of the ionic conductor library



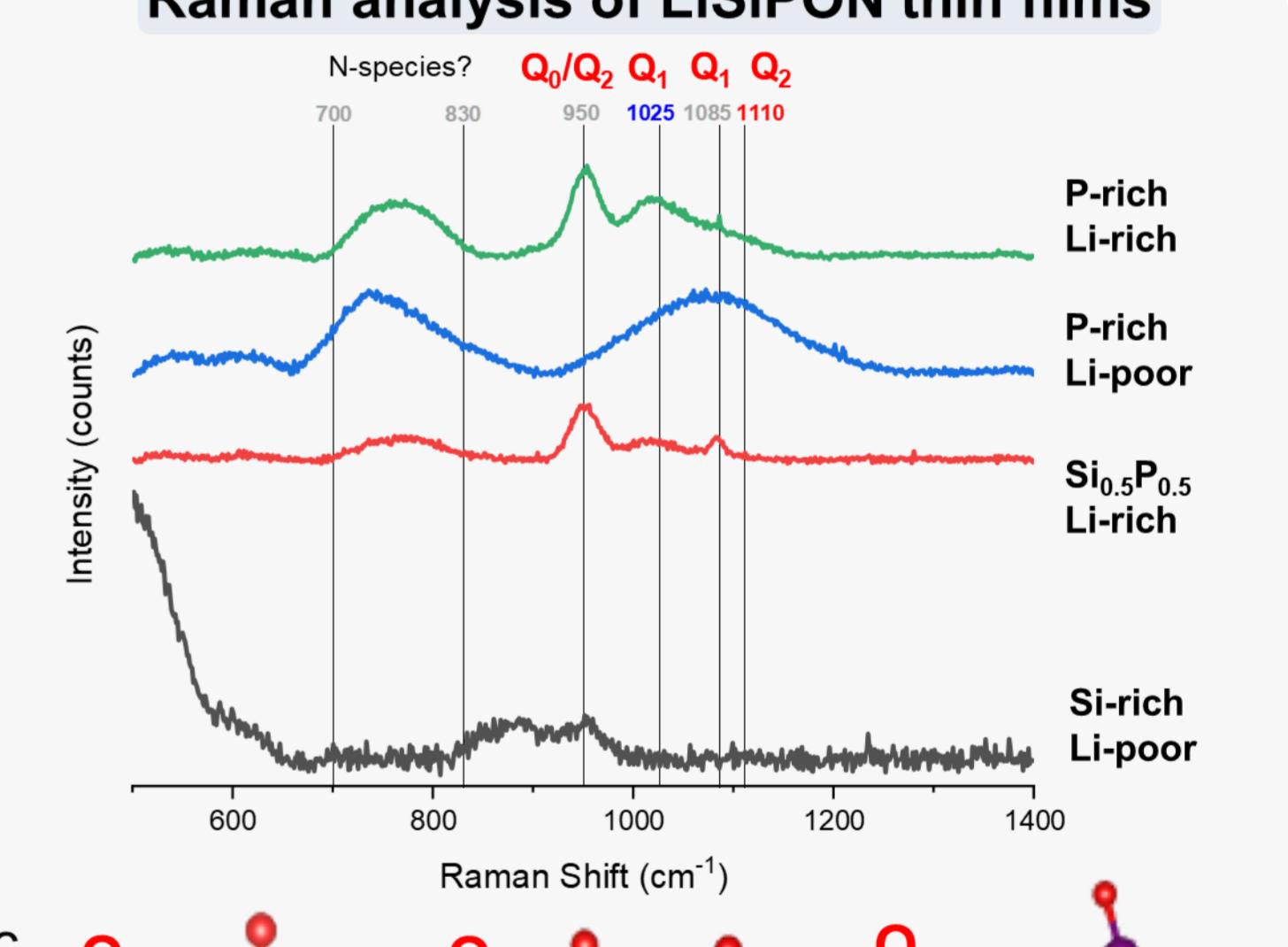
Ionic conductivity mapping



Raman analysis of powders

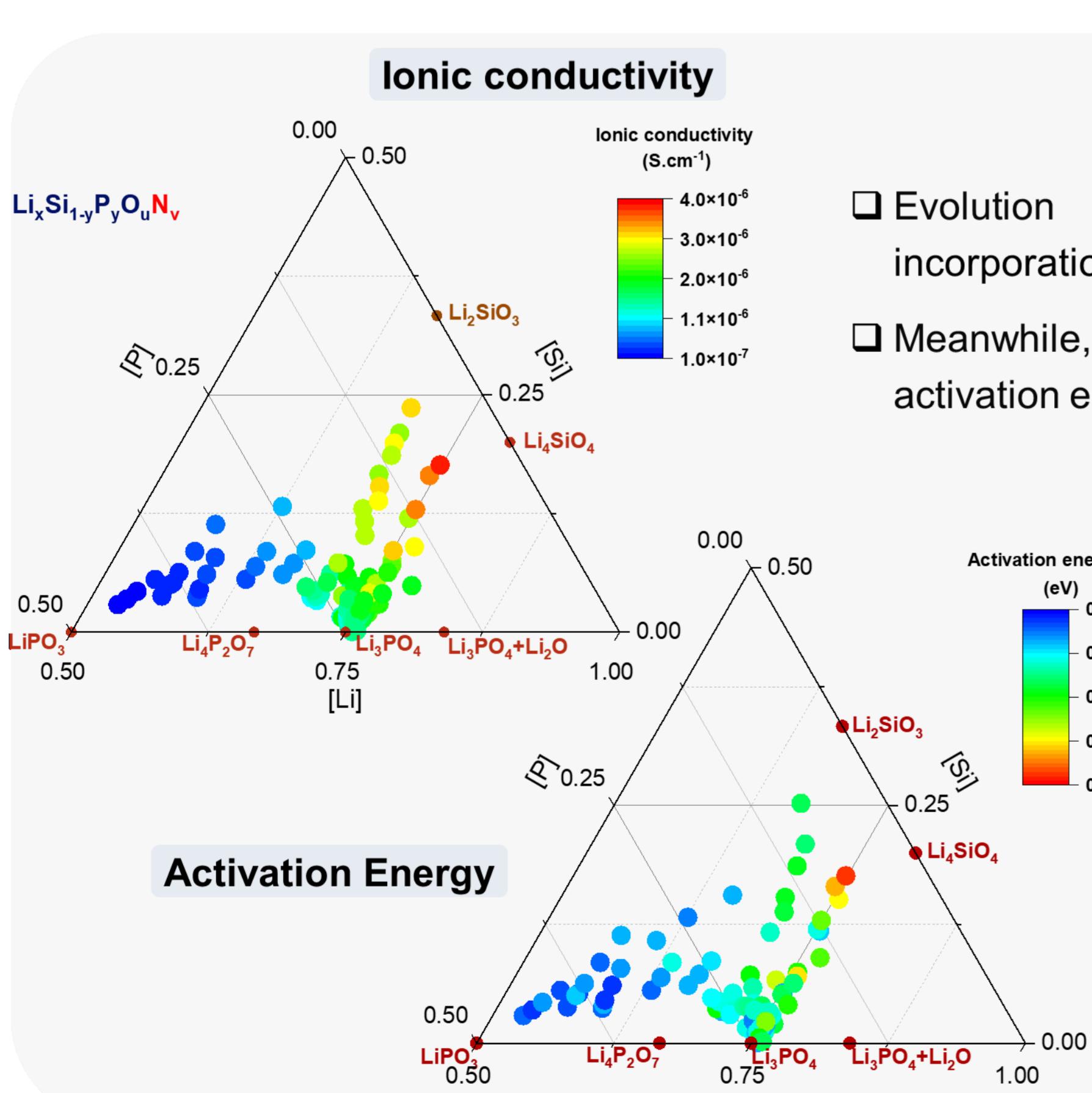


Raman analysis of LiSiPON thin films



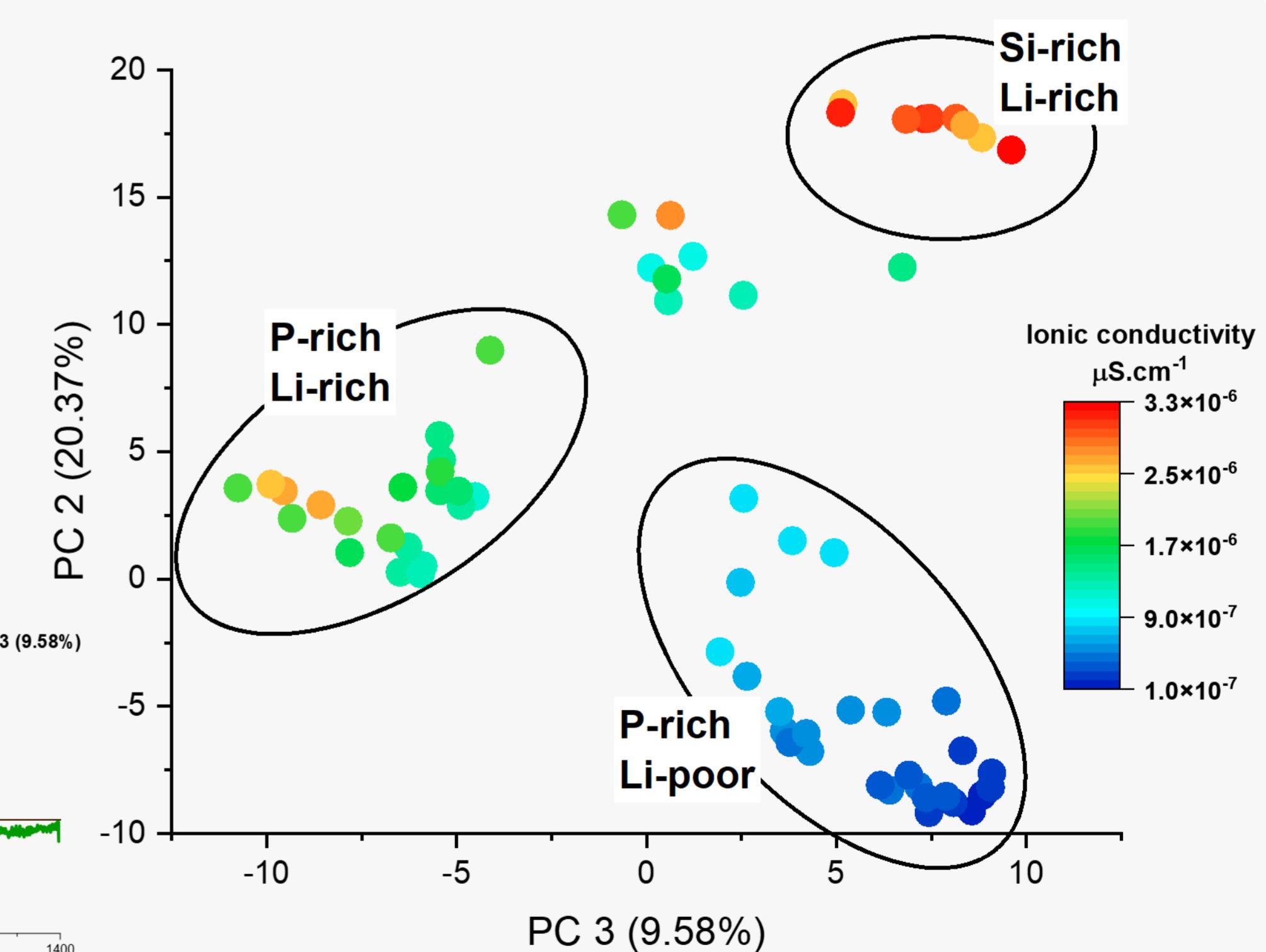
□ Evolution of vibrational modes, attributed to specific building blocks within the amorphous LiSiPON

Thorough understanding by correlative analysis



PCA analysis of LiSiPON Raman spectra

- Score plots, displaying 'meta-spectra' (PCs) within the material library
- Loading plot, showing LiSiPON distribution within the 'meta-spectra' (PCs)



5. Take Home Message

- All-in-one HTS platform for characterization of solid ionic conductors
- Screening of chemical – structural – functional properties of LiSiPON thin films library deposited by combinatorial sputtering
- This correlative study provides new insight about the role of Li and Si incorporation in oxynitride phosphorus media, in terms of material structure and ionic conductivity

Acknowledgments

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