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In recent years, lithium-ion batteries, used in a variety of electronic devices and vehicles, have been facing challenges in terms of sustainability and lithium availability, thereby necessitating a better understanding of their ageing mechanisms. To this end, the ANR Micro-Q-Li project, conducted in partnership with the French Institute of Light and Matter, developed an enhanced LIBS imaging prototype that achieves a spatial resolution of 1.5  $\mu\text{m}$ , exceeding the limitations of traditional analytical techniques for lithium imaging.

## Pushing back the limits of LIBS imaging to locate lithium

In recent years, lithium-ion batteries have become part of our everyday lives: in our telephones, laptops, handheld tools and, more recently, our electric and hybrid vehicles. However, these batteries have a limited lifespan, and the challenges of sustainability, particularly with regard to the availability of lithium resources, make it necessary to seek to improve their longevity. This requires **a detailed understanding of the ageing mechanisms at play** and knowledge of the exact location of lithium, at the micron scale, in aged electrodes.

Few analytical techniques enable lithium imaging with **sufficient sensitivity and spatial resolution**. The same is true of LIBS (Laser Induced Breakdown Spectroscopy) imaging, which, while highly sensitive to lithium, remains limited by a lateral resolution of 10  $\mu\text{m}$ , insufficient to resolve the microstructure of electrodes [1].

In order to overcome these limitations, [the ANR Micro-Q-Li project](#), conducted in partnership with [the French Institute of Light and Matter](#), set out to reduce the lateral resolution of the technique by a factor of 10. To achieve this, a prototype LIBS imager, illustrated in Figure 1, was designed and built during the project. It delivered **a resolution of 1.5  $\mu\text{m}$**  in the most favorable cases.

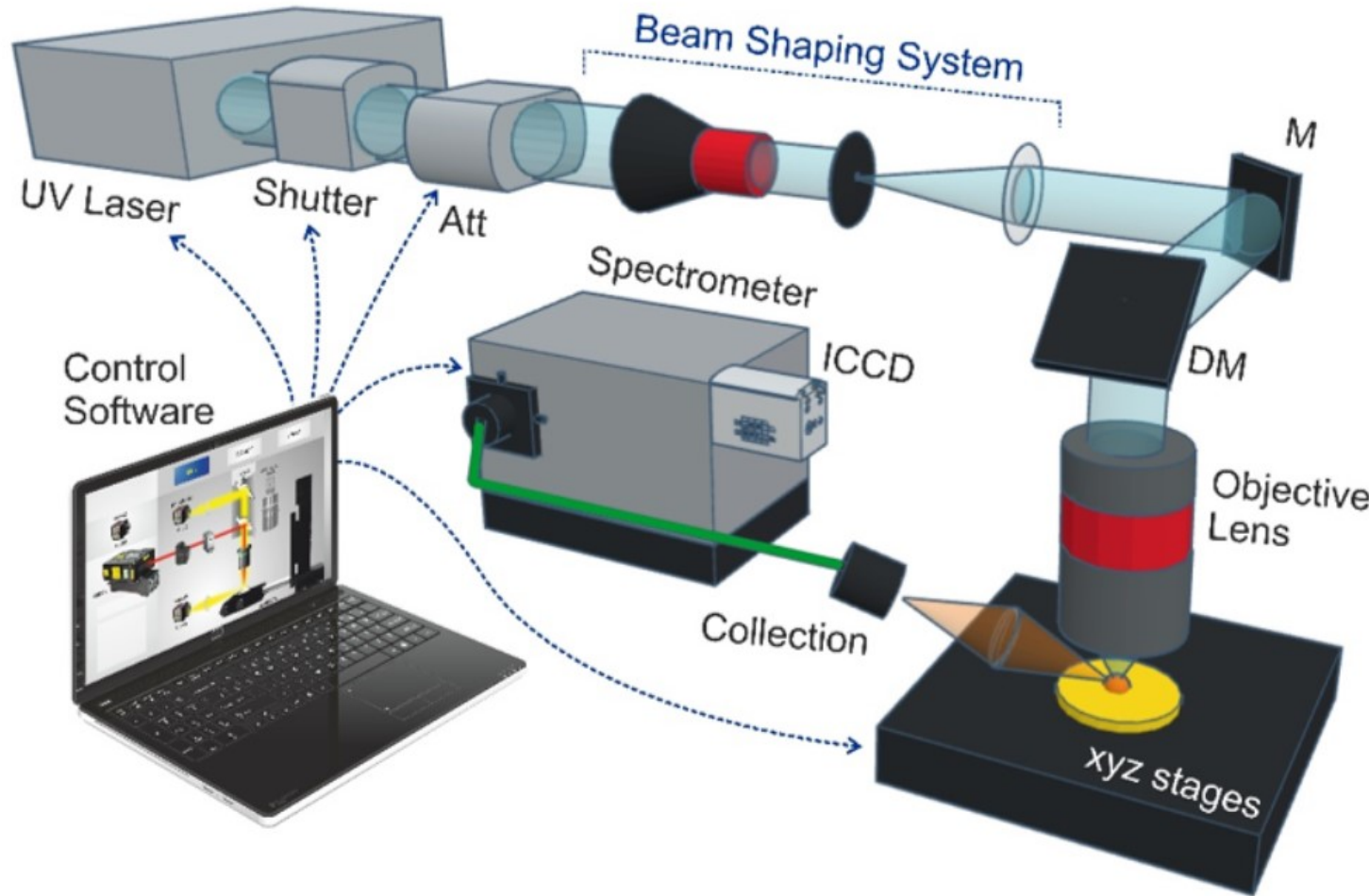


Figure 1: Illustration of the LIBS imager prototype. Figure adapted from [2]

## An imager that follows the evolution of lithium in electrodes

This prototype was used to image NMC (Nickel Manganese Cobalt)-type positive electrodes extracted from batteries in different states of charge, with **a lateral resolution of 2  $\mu\text{m}$** . The results shown in Figure 2 confirm the expected performance and value of this type of imager:

- firstly, its resolution is sufficient to identify the spherical NMC grains forming the active layer of the electrode that intercalates and de-intercalates the lithium;
- secondly, it enables observation of **the decreasing abundance of lithium** as a function of **the battery's state of charge** (the positive electrode contains all the cyclable lithium when the battery is discharged and becomes progressively depleted of this element as the battery is charged).

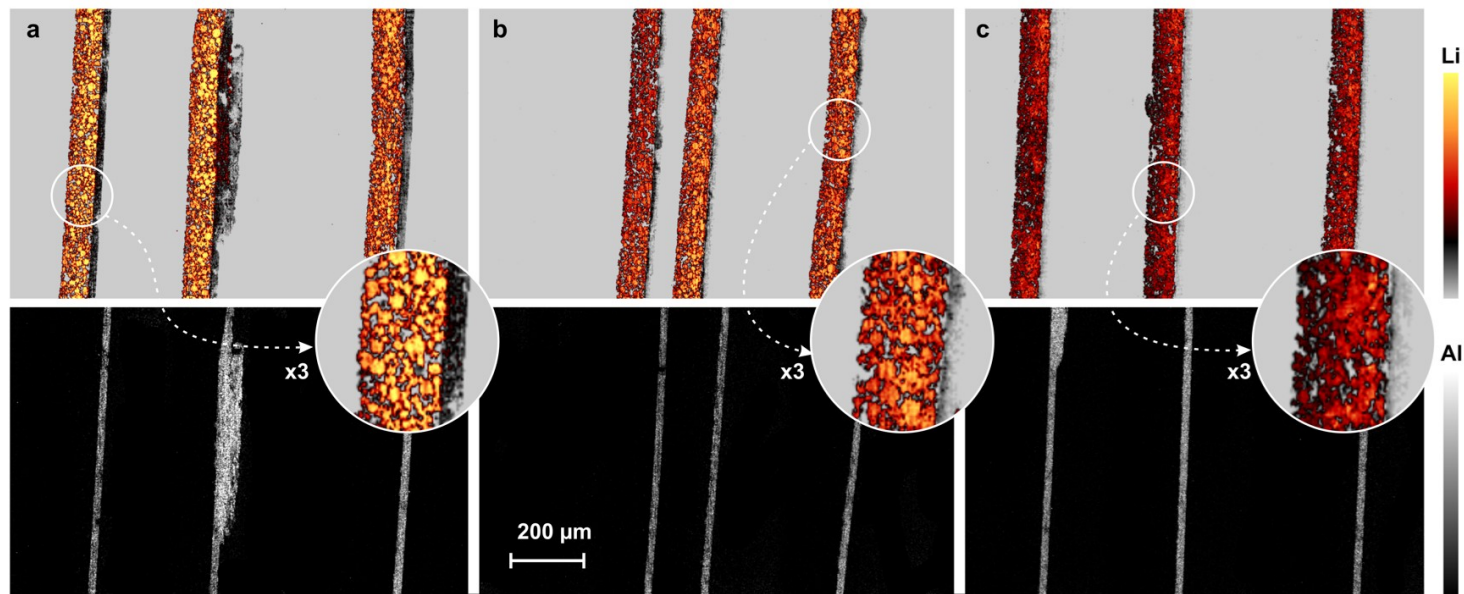


Figure 2: Abundance maps for lithium (top) and aluminum (bottom) in NMC electrode sections at different battery states of charge [2]. (a) 0% (b) 50% (c) 100%

## A better understanding of battery ageing mechanisms

The imager was also used to characterize electrodes from commercially available cells that have undergone two different ageing modes: calendar (full charge, high temperature) and cycling (fast charge, low temperature) [3]. The results made it possible to identify **the ageing mechanisms** responsible for performance losses, some of them known (lithium trapping in the negative electrodes through the formation of a solid-electrolyte interphase, lithium plating, grain disconnection), and others unexpected, such as lithium trapping in the copper collector of the negative electrode.

## Promising prospects for the future of batteries

During the course of the project, problems were also identified in terms of sample preparation and the management of sample transfer without contact with air. Lastly, the development of this imager opens up interesting prospects for lithium batteries, both **for improving the longevity of existing technologies** and **for developing new ones**.

### References :

- [1] L. Jolivet, M. Leprince, S. Moncayo, L. Sorbier, C.-P. Lienemann, V. Motto-Ros, Review of the recent advances and applications of LIBS-based imaging, *Spectrochimica Acta Part B* 151 41–53 (2019),  
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>> <https://doi.org/10.1016/j.sab.2024.107047>

[3] J. Fernandes, Caractérisation du lithium : Applications aux systèmes de stockage électrochimiques, thèse de doctorat de l'Université Claude Bernard Lyon 1, soutenue le 19/12/2024

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