

Analyse Li-ion battery anodes with the inVia™ confocal Raman microscope

Materials science

Li-ion battery anodes consist of a variety of components and materials, including conductors, carbon, and binders. Different types of carbon are used in anodes to fulfil different purposes, such as participating in intercalation reactions with lithium ions, and providing conducting paths for charge carriers. They are typically produced by pyrolysis of organic, polymer or hydrocarbon precursors.

The different physical properties of carbon forms—such as their electro-conductivity, BET (Brunauer–Emmett–Teller theory) surface area (or porosity at the nano level) and electric capacity—are determined by their local structures. These structures are characterised by the amount of long range order of the planar hexagonal network, the amount of embedded amorphous areas, and the extent of cross-linking. The structure depends on the composition of the precursor and pyrolysis conditions.

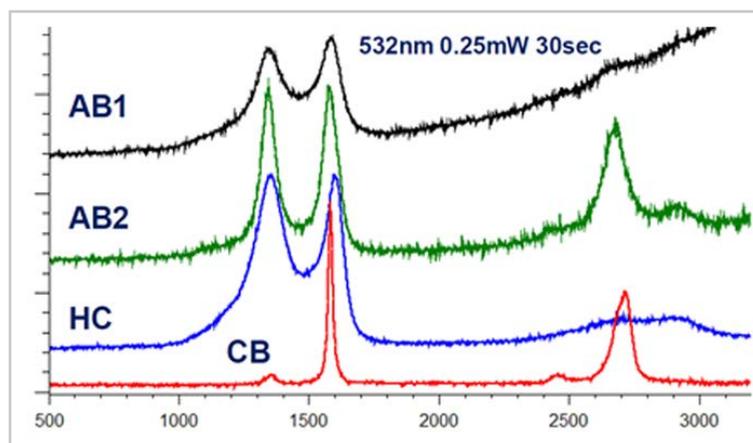
It is therefore vital to be able to rapidly and easily analyse the composition and structure of anodes. The inVia confocal Raman microscope is ideal for locating, discriminating, and quantifying the different forms of carbon present in anodes, even those with subtle variations in structure.

Analysis of carbon materials in anodes

The following example is of the analysis of these typical carbon forms:

- Acetylene black (AB)
- Hard carbon (HC)
- Carbon black (CB)

These materials have broadly similar Raman spectra, but can be identified by the intensity ratio between the carbon D- and G-bands, and the width of the G-band.



Raman spectra for four different carbon samples, two acetylene blacks (AB), hard carbon (HC), and carbon black (CB). The D-band is at $\sim 1350 \text{ cm}^{-1}$, the G-band at $\sim 1600 \text{ cm}^{-1}$

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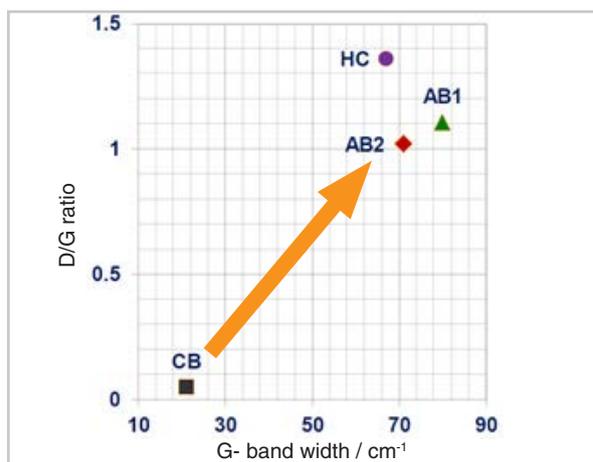
The process involved in analysing these materials is:

- collect spectra under the same conditions
- fit curves to D-G-band region
- plot D/G intensity ratio vs G-band width (full-width, half-maximum)(G).

Interpreting the data:

- higher D/G ratio implies shorter correlation lengths of hexagonal planar sp^2 domains, (smaller crystallites)
- a wider G-band indicates local disorder in the sp^2 layer

These two parameters can be plotted to indicate the level of disorder (right). Differences as small as 0.1 in D/G ratio, or 10 cm^{-1} in G band width can easily be detected.

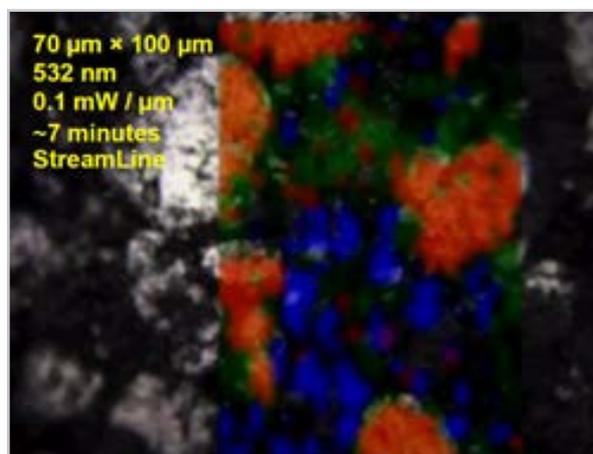


Intensity ratio of the D-band and G-band plotted against width of the G-band. The orange arrow indicates the progression from order to disorder.

Creating chemical images of anodes

Images generated using inVia's StreamLine™ technology can be used to reveal the distribution of these materials in the anode. The images can also be used to determine the relative amounts of material and statistical parameters such as average particle size and (in)homogeneity of the distribution across the layer.

Renishaw's LiveTrack™ automated focus tracking technology enables the easy analysis of electrode surfaces even if they are uneven or rough.



StreamLine Raman image revealing: graphite (red); acetylene black (blue); and hard carbon (green). The relative ratios are: graphite (13%); acetylene black(19%); hard carbon (77%).

The comprehensive analysis tool

These results illustrate the ease with which you can use Renishaw's inVia Raman microscope to find and distinguish these different carbon materials, and to compare anode samples with different performances.

inVia. The ideal Raman battery analysis tool

- Research grade Raman microscope
- High sensitivity to detect traces of material
- High confocality to scrutinise small details
- A range of rapid mapping and imaging solutions
- Automation options, such as triggering of data acquisition by external systems (e.g. potentiostats)
- Raman-compatible electrochemical test cells available



The Renishaw inVia confocal Raman microscope

Renishaw. The Raman innovators

Renishaw manufactures a wide range of high performance optical spectroscopy products, including confocal Raman microscopes with high speed chemical imaging technology, compact process monitoring Raman spectrometers, structural and chemical analysers for scanning electron microscopes, solid state lasers for spectroscopy and state-of-the-art cooled CCD detectors, for both end-user and OEM applications.

Offering the highest levels of flexibility, sensitivity and reliability, across a diverse range of fields and applications, the instruments can be tailored to your needs, so you can tackle even the most challenging analytical problems with confidence.

A worldwide network of subsidiary companies and distributors provides exceptional service and support for its customers.

Please visit www.renishaw.com/carbon for more information.