Closed vessel used for Raman analysis in inert atmosphere

LIBcell

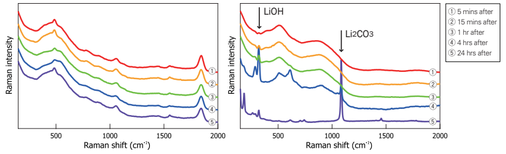
Environmental cell for inert atmosphere analysis

The environmental sample cell is fabricated from corrosion resistant stainless steel fitted with a 1mm-thick quartz observation windowv. Cell construction is gas tight providing a secure, inert gas environment for Raman analysis. Sample loading is easily performed in a glove box before loading the environmental cell onto the microscope stage. (Patent pending)

Actual size

The size of the sample chamber is 10mm diameter by 1.5mm deep. The sample chamber region of the LIBcell can accommodate a range of sample thicknesses. For optimum analysis, position the sample such that it is close to the inner quartz surface, using spacers as necessary.

Environmental Cell Integrity



Lithium metal in the LIBcell

Lithium metal exposed to air

The long-term integrity of the LIBcell was determined using Lithium metal samples. When lithium metal is exposed to air the surface quickly is converted to a dull silver-gray color. In the presence of air the metal surface is converted to LiOH and then to Li_2CO_3. The spectra on the left show no change in the state of lithium after 24 hrs in the LIBcell under inert gas conditions.

Nanophoton Corporation

Photonics center #321, 2-1 Yamadaoka, Suita, Osaka 565-0871 JAPAN

TEL: +81-6-6878-9911 FAX: +81-6-6878-9912

E-mail: info@nanophoton.jp

Local distributor

2011.09

nano photon

Laser Raman Microscope

Lithium-ion Battery Applications

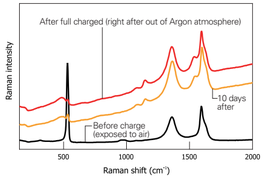
www.nanophoton.jp

In the rechargeable battery industry, Raman spectroscopy is now recognized as an important analytical technique, as it allows for the measurement of chemical bonding, composition, and crystalline state of a sample.
Raman microscopy extends this vital analytical technique by having the ability to observe and analyze selected regions of a sample as small as just a few hundreds of nanometers in diameter. This provides investigators intimate knowledge of localized reaction sites within a sample.
Following are 3 examples of how Raman microspectroscopy adds significant value to battery research.

Analysis in an inert atmosphere (LIBcell is used as a vessel)

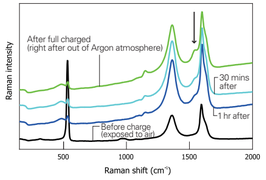
Raman microspectroscopy can monitor and measure the condition of a charged anode, as shown in the left hand spectra. By performing such measurements in a LIBcell the reduction status of the electrode can be monitored over time while eliminating possible degradation due to environmental conditions.

Measurement with LIBcell



Measurement of charged electrode in Argon atmosphere in a LIBcell. Spectra illustrate that no electrode deterioration occurs over a 10-day time period following a charging cycle.

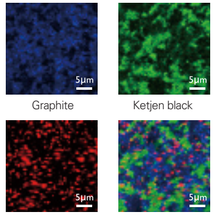
Measurement exposed to air



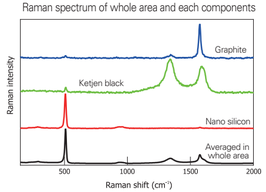
Measurement of charged electrode exposed to air. This graph illustrates spectral changes reflecting electrode degradation resulting from exposure to air.

Observation of component distributions (Raman imaging)

The distribution of a range of components observed by Raman imaging of the sample surface are shown on the right hand side. The each spectrum defines component of the battery, even when regions analyzed show a mixture of individual components. Raman microspectroscopy allows focus of the laser beam to a small spot making it possible to observe and identify sub-micron distributions of silicon scattered throughout the silicon anode.



Raman spectrum of whole area and each components

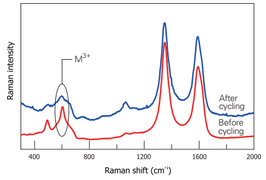


The distribution of each component can be observed by color coding spectra of various battery components, as shown in the graphs above.

Observation of crystal structure (comparison to XRD)

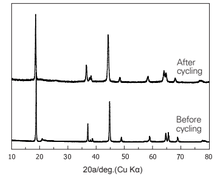
Cathodic deterioration occurs as a result of disorder in crystal structure. Normally crystalline structures have been analyzed using x-ray diffraction, a method that gives bulk information, such as crystalline structure with long periodicities at the level of a few tens of micrometers. In contrast, Raman microspectroscopy permits the evaluation of crystallinity changes at the micrometer level. Furthermore, Raman spectroscopy requires only few minutes to detect and characterize changes in local crystalline structure.

Raman spectroscopy



Comparison of spectra of before and after the charging cycle of cathode. The graph shows the broader peak of M^{3+} which explains the crystalline change of cathode after the cycle.

XRD



Examination with XRD of the same sample. Change of crystal structure is observed by the slight change of peak broadness.

*These samples are provided by research team of Associate Professor Shinichi Komaba in Tokyo University of Science.