Nanophoton Products

Special Accessories for Raman Analysis of Li-ion Battery Electrodes

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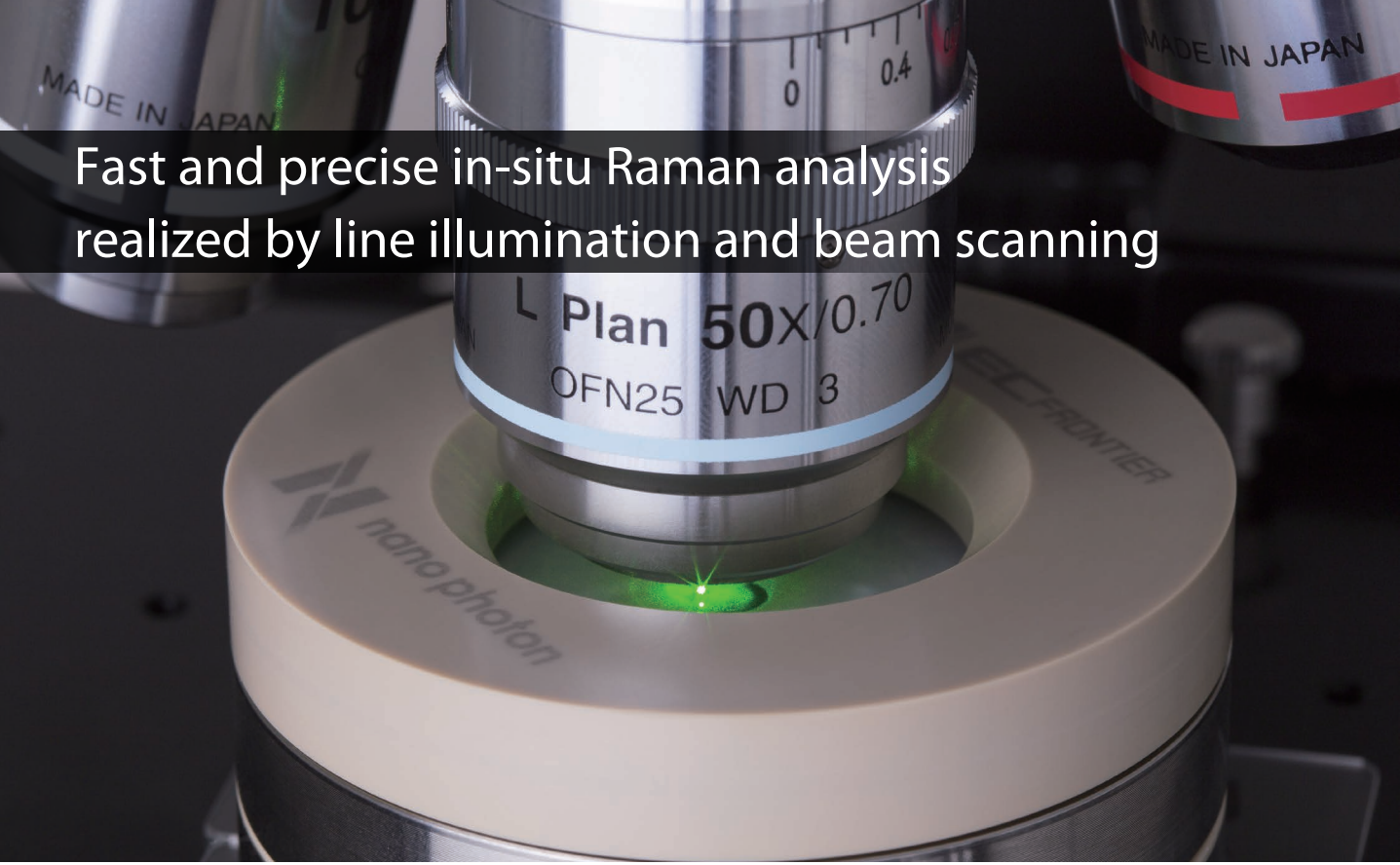
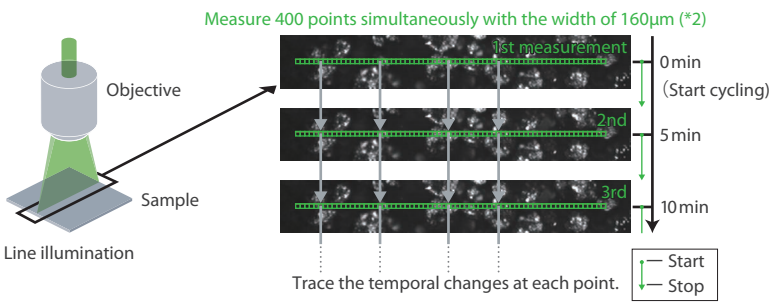


Fast and precise in-situ Raman analysis realized by line illumination and beam scanning

X-line measurement mode is incorporated in laser Raman microscope RAMANtouch which uses only one line of line illumination to measure. 400 spectra of the linear region from approximately 80μm to over several hundred μm (*1) can be obtained by one-shot exposure. Combine with the use of LIBcell charge, multi- point analysis is executable with charging and discharging being maintained after a short time interval.

In-situ analysis using X-line measurement mode

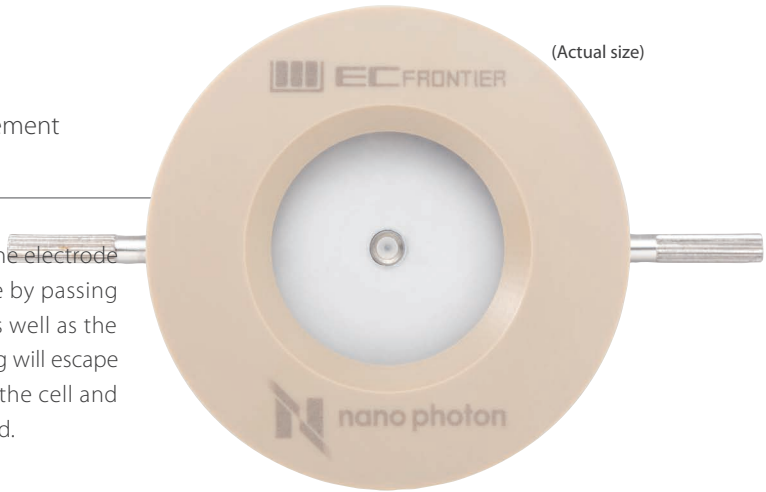


*1 The length of line illumination varies according to the magnification of objective.
*2 An example of a 50x objective lens being used.

Product Lineup

Charge/discharge cell for in-situ Raman measurement

LIBcell charge



(Actual size)

LIBcell charge is a cell for in-situ Raman analysis of the electrode surface. It measures the opposite electrode surface by passing laser through the holes of the counter electrode as well as the separator. Gas generated by charging and discharging will escape from the observation part located at the center of the cell and interference that affects the measurement is restrained.

LIBcell charge specification

Size : φ64 × 31mm (Electrodes not included) **Weight :** 600g **Material :** PEEK/SUS316 **Window :** φ31 × t1mm quartz (t1.3mm at measurement area) **Size of electrodes :** OD16mm/ID4mm(For observation), OD16mm/ID4.5mm(Separator), OD16mm/ID5mm(Counter) **Electrode for lead :** SUS316/20mm **O-ring :** Perflour/Butyl

Closed vessel for Raman analysis in inert atmosphere

LIBcell

(Actual size)

The environmental sample cell is fabricated from corrosion resistant stainless steel fitted with a 1mm-thick quartz observation window. 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. (Patented)

LIBcell specification

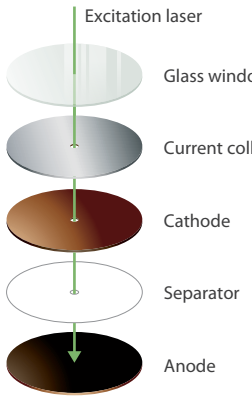
Size : φ80 × 12mm (Thumbscrew not included) **Weight :** 400g **Material :** SUS303 **Window :** φ45 × t1mm quartz **Space for sample :** φ10 × H1.5mm **O-ring :** Viton

* Long W.D. objective or correction collar objective is necessary for both products when more than 50x magnification is requrired for measurement.

In-situ Raman imaging analysis of graphite anode

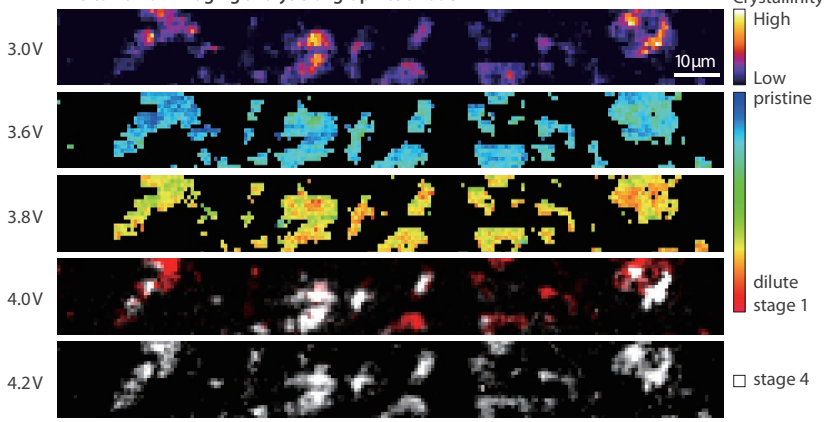
Analysis by the use of LiBcell charge and Raman imaging visualizes the difference in how the progress status of charging reaction varies according to the locality. The result of in-situ Raman imaging of graphite anode is shown in the lower right image. Charging reaction is known to be in progress from the indication of high crystallinity due to the discharge status. Various charging and discharging process can be examined by in-situ with the change in electrode material and electrolyte solution, shift between anode and cathode along with the adoption of LIBcell charge.

Structure of LIBcell charge



Measure the electrode surface at the bottom by making small holes on the separator as well as the counter electrode and let laser pass through. The periphery of the opening which has a higher efficiency of charging and discharging reaction is targeted for analysis.

In-situ Raman imaging analysis of graphite anode



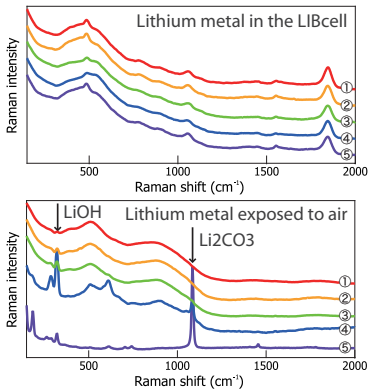
532nm / 50x, 0.70NA / 400 x 20 pixel / 14 min

It took about 14 minutes to obtain one Raman image. Highly time-resoluved in-situ analysis would be possible using X-line measurement mode.

Raman imaging of silicon anode in inert atmosphere

The Raman images below show the comparison of silicon-graphite composite anode before and after charge. Anode before charge was measured in the air, while that of after charge was packed into LIBcell in a globe box and measured under inert atmosphere. It could be confirmed that almost all of crystalline-silicon was changed into amorphous state by its lithiation process during charging. Diffraction-limited spatial resolution of RAMANtouch was fully performed by using objective with correction collar, thus the distribution of active materials could be clearly visualized.

Integrity test of LIBcell airtightness

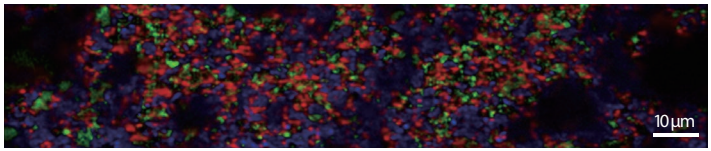


① 5 mins after
② 15 mins after
③ 1 hour after
④ 4 hours after
⑤ 24 hours after

The spectra of Lithium metal in the LIBcell show no change after 24 hours under inert gas conditions.

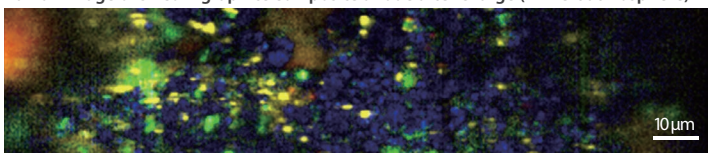
The size of the sample chamber is 10mm diameter by 1.5mm deep. The sample chamber region of the LIBcell can accommodate a range of sample thicknesses. For optimum analysis, position the sample such that it is close to the inner quartz surface, using spacers as necessary.

Raman image of silicon–graphite composite anode before charge



532nm / 100x, 0.85NA / 400 × 100 pixel / 35 min

Raman image of silicon–graphite composite anode after charge (in inert atmosphere)



532nm / 100x, 0.85NA / 400 × 100 pixel / 35 min

■: Crystalline Silicon ■: Amorphous Silicon ■: Graphite ■: Ketjen Black

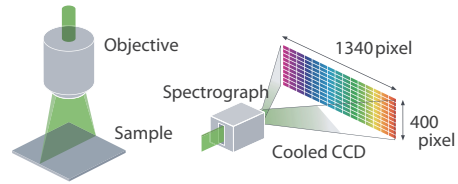
Laser Raman microscope developed by Nanophoton

Laser Raman microscope

RAMAN touch

Clearly resolved Raman image of a small-scale structure can be obtained by diffraction-limited spatial resolution of RAMANtouch. Patented line illumination and dedicated laser beam scanning enables ultra-fast Raman imaging without moving the stage, which is best suited for the measurement using LIBcell and LIBcell charge.

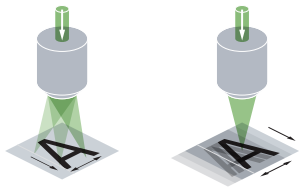
400 spectra simultaneous detection by line illumination



Line illumination　400 spectra detection

Raman light generated from the line-illuminated area is divided into 400 points, while each light is being expressed with 1340 points.

Vibration-free imaging by laser beam scanning



Laser beam scanning
RAMANtouch

Stage scanning
Competitors

Ultfa-fast and precise imaging is possible even when the sample is on the fixed stage or in the LIBcell and LIBcell charge.

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LIBcell charge developed jointly with
EC Frontier., Inc.

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