Title

: CalTestBed - OnTo Technology - Characterization of Recycled Cathode and Anode Particles as a Function of Process Step

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Cooperative Research and Development Agreement (CRADA) Final Report

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In accordance with Requirements set forth in the terms of the CRADA, this document is the CRADA Final Report, including a list of Subject Inventions. It is to be forwarded to the DOE Office of Scientific and Technical Information upon completion or termination of the CRADA, as part of the commitment to the public to demonstrate results of federally funded research.

Parties to the Agreement: ONTO Technology LLC and Lawrence Berkeley Lab

CRADA number: FP00012070

CRADA Title: CalTestBed - OnTo Technology - Characterization of Recycled Cathode and Anode Particles as a Function of Process Step

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Sponsoring DOE Program Office(s):
N/A

LBNL Report Number:
[PI to complete]

OSTI Number:
[SPO to complete]

Joint Work Statement Funding Table showing DOE funding commitment:

DOE Funding to LBNL	$300,000
Participant Funding to LBNL	0
Participant In-Kind Contribution Value	$136,751
Total of all Contributions	$436,751

Provide a list of publications, conference papers, or other public releases of results, developed under this CRADA:
(Publications must include journal name, volume, issue, Digital Object Identifier)
None

Provide a detailed list of all subject inventions, to include patent applications, copyrights, and trademarks:
(Patents and patent applications are to include the title and inventor(s) names. When copyright is asserted, the Government license should be included on the cover page of the Final Report)
None

Executive Summary of CRADA Work:
Onto Technology carries out advanced Li-ion battery recycling for industry and. has the only patented technology to produce manufacturing quality electrode materials from recycled batteries. The technology can resolve ongoing cost problems for the industry in life-cycle and conservation of critical materials. Under the Cal TestBed program, Onto Technology teamed with principal investigators at Lawrence Berkeley National Laboratory, who performed tests to evaluate the electrochemical performance of refurbished electrode materials. Other tests to determine structure and morphology of the refurbished materials carried out at LBNL included X-ray diffraction (XRD) and scanning electron microscopy (SEM).

Summary of Research Results:
Results of electrochemical testing and physical characterization carried out at LBNL were provided to Onto Technology. Examples are given below (data approved for release by Onto).

LBNL received a sample, labeled 2 V-D (for vac, then deactivate) from OnTo that looked like the photo in Fig. 1 before and after disassembly.

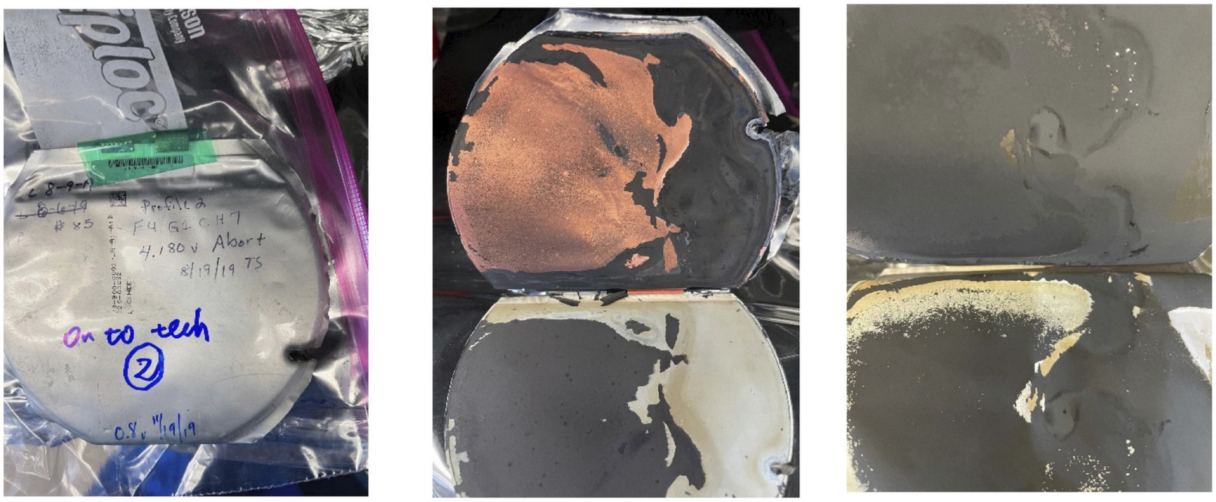


Figure 1. *Left* Full cell. *Middle* Anode. *Right* Cathode. There is a notch on the full cell near the edge, just below the equator. The anode peeled off the separator. The cathode peeled so that half was left on the current collector and the other half on the separator.

LBNL was asked to test the anode and cathode individually in symmetric cells. Since the sample was split along the middle of the cathode, we were unable to test it in symmetric cells. Samples near and far from the notch were collected and electrochemical impedance spectroscopy was carried out. The impedance near the notch was around 70 $ohm \cdot cm^2$ and the impedance far from the notch was around 30 $ohm \cdot cm^2$.

A second sample labeled *3 – NV-D*, (for no vac, then deactivate) was tested at spots near and far from a notch found in the cell. The impedance of the anode near the notch showed much more ohmic loss as if detached from the current collector and about twice as much charge transfer resistance as the part far from the notch. The impedance of the cathode was about the same for the samples near and far from the notch. In half-cells, the anodes started with the same capacity but the anode near the notch cycled better than the anode far from the notch. The cathode half-cells cycled about the same but with significant impedance between charge and discharge.

Three powder samples labeled *4a Baseline*, *4b Hydrothermal*, and *4c Heat* 600^oC were received. LBNL made electrodes and coin cells and collected the charge and discharge voltage curve data for the three samples (Figure 2) and found that the baseline discharge curve is slightly above the Heated 600^oC curve, which is above the hydrothermal curve. The baseline cycled with very little capacity fade where the other two cycled with about the same capacity fade of about 15 % after 100 cycles.

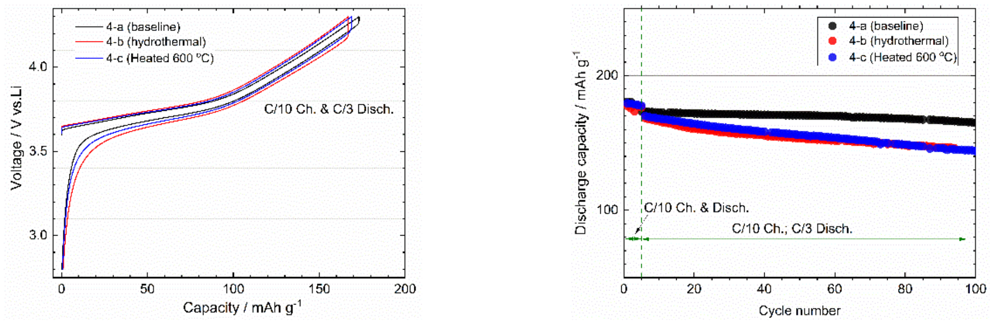
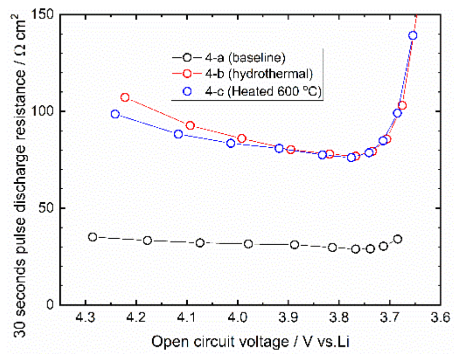


Figure 2. (Left) Charge and discharge voltage curves of three samples: *4a baseline*, *4b Hydrothermal*, and *4c Heated 600 ºC*. (Right*)* Cycle capacity of the three cells *versus* cycle number.

A High Pulse Power Characterization test (HPPC) was performed on the cells to estimate the resistance due to 30-second current pulses of 5C. The baseline resistance was well below that of the other two, which were

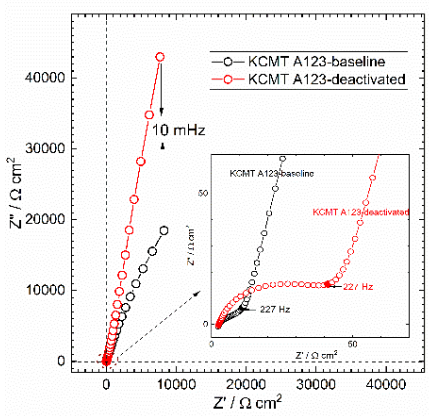


indistinguishable from each other (Figure 3).

Figure 3. HPPC data of cells made from the three powders labeled *baseline*, *hydrothermal*, and *heated 600ºC*.

Sample 5 consisted of two different commercial materials. 5 A was a cathode from a cell that was opened in air and transferred to a bottle under argon; 5 B was an anode from a cell that was also opened in air and transferred to a bottle under argon. Cells made from the cathodes displayed a range of capacities from 3.15 to 3.6 mAh/cm2 and about 25% fade after 100 cycles. The anodes from the second cell delivered essentially no capacity.

Sample 6 consisted of anodes from two different cells: a *baseline* cell and a *deactivated* cell. The interfacial impedance of the deactivated cell was much greater than the baseline cell, as seen in the inset of Figure 4. The reversible capacities obtained at C/10 were about the same. (Note: identities



of manufacturers have been removed).

Figure 4. Impedance of anode half cells from sample 6, labeled *baseline* and *deactivated*.