



Short communication

Microscopic in-operando thermography at the cross section of a single lithium ion battery stack

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ABSTRACT

A cell design to perform microscopic in-operando thermography across the interface anode – separator/electrolyte – cathode of a single lithium ion battery stack has been developed. A 3-electrode Swagelok® cell is adapted to enable the use of microscopic thermography during battery cycling. The developed measurement setup has been successfully tested using LiCoO₂ as cathode, LiPF₆ salt containing organic electrolyte and graphite as anode. The measurement results reveal differences in the heat generation of the individual components of the single battery stack. Furthermore, inhomogeneity in the electrodes can be detected and the heat transport on the micro scale can be observed.

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1. Introduction

The widening of applications for lithium ion batteries is dragging due to several disadvantages, e.g. concerning safety and reliability caused by heat generation during charging and discharging. Fundamental knowledge of heat generation and transfer on the micro and macro scales is obligatory to optimize the material, design and management of batteries.

The majority of publications concerning the heat generation in lithium ion batteries is focused on integral measurements (accelerated-rate calorimetry [1,2], isothermal heat conduction calorimetry [3–5], measurement of the surface temperature [6,7]) of prismatic, cylindrical or coin cells (see e.g. [8]). Recently, spatially resolved temperature measurements have been published [9–11]. However, the achieved spatial resolution is much larger than the dimensions of the individual battery components of anode, separator and cathode.

Actually, the heat generating mechanisms and the material properties distinctly differ between the individual components of a lithium ion battery stack [12].

According to this, in-operando measurements of the temperature distribution inside a single battery stack can reveal:

- Information concerning the heat transfer on the micro scale
- A fundamental understanding of the local heat generation as a function of the C-rate and the SoC
- Complementary information to kinetics and thermodynamics.

Therefore, the authors developed a cell design that offers the following possibilities:

- Electrochemical measurements in 3-electrode configuration
- Cross sectional observation of a single battery stack using microscopic thermography during the electrochemical measurements.

For the validation of the experimental setup a well investigated electrode system is used.

2. Experimental

Commercially available composite electrodes (MTI) containing active material (LCO, C), conductive additives and binder on a current collector were investigated in a 3-electrode arrangement. The reference electrode was metallic lithium. The separator was a borosilicate glass-microfiber filter (Whatman).

Commercially available LP40 (BASF) was used as electrolyte. The assembly of the cell was carried out in an argon filled glove box (MBraun). The electrochemical experiments included constant current charge–discharge cycles. Therefore, a potentiostat/galvanostat with a frequency response analyzer VMP3 (BioLogic) was used.

The thermographic device ImageIR-8480® (INFRATEC) extended with a microscope lens offers the following specifications:

- Spatial resolution: 5 μm
- Temperature resolution: <0.02 K
- Spectral range: MWIR (2–5 μm)
- Frame rate: 200 Hz.

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The thermographic measurements were carried out simultaneously to the electrochemical experiments.

3. Results and discussion

3.1. Cell design

A typical cell design used in battery research is adapted from products of a pipe coupling manufacturer and called Swagelok® cells. The cell design presented here was developed by adapting a 3-electrode Swagelok® cell for the use of microscopic in-operando thermography. A cross sectional view of the developed cell design is presented in Fig. 1. The cell body (1), made of PFA, offers an inlet for a reference electrode (2). A window (3) made of sapphire is implemented into the cell body which is transparent in a range of near UV to IR as well as chemically inert and mechanically stable. Special stamps (4) are developed consisting of three main parts, the top region (4.1), the middle region (4.2) and the outer region (4.3). The top region (4.1) is a flattened cylinder made of PEEK and stainless steel or aluminum as electrical contacts. The middle region (4.2) offers mechanical flexibility and electrical contact between the top region and the outer region. This ensures a constant pressure and a reproducible electrical contact within the battery stack. The configuration reflects the conditions inside commercial batteries (multistacked or wound battery as 18650) on the scale of single electrodes unaffected by their respective cell geometry or processing (details are described e.g. in [8]). The setup is strictly focused on the heat generation which is exclusively associated with the electrochemical processes and unaffected by the cell geometry. The outer region (4.3) enables the sealing of the cell and the electrical connection to the potentiostat/galvanostat.

As reference electrode the tip of a flattened lithium metal wire (<0.5 mm in diameter) is embedded in a double-layer separator peripherally at the electrodes. The influence of type, position and geometry of the reference electrode in such test cells is currently extensively discussed [13,14]. A significant influence of the position of the reference electrode is reported for precise impedance measurements especially at frequencies >1 kHz. However, this effect may be neglected in view of charging–discharging experiments which are in the range of at least some 10 s.

Fig. 2 shows the cell design after being assembled in the glove box and mounted on a 3D micro positioning system which allows an exact focusing. Furthermore, this enables an accurate scanning of the sample without moving the thermographic device and without risking defocusing.

3.2. In-operando measurements

To evaluate the proper functioning of the developed cell design, charging–discharging experiments are performed. Fig. 3 shows the

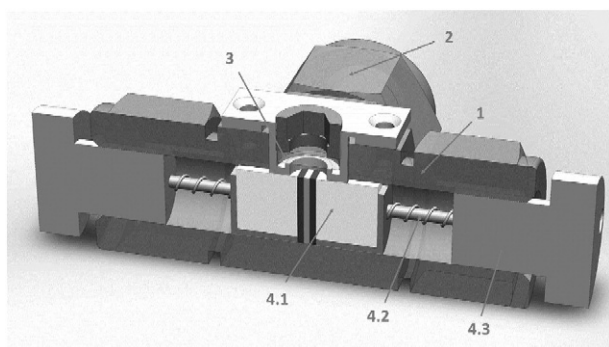


Fig. 1. Cross sectional view of the developed cell design, certain parts are indicated by numbers and explained in the text.

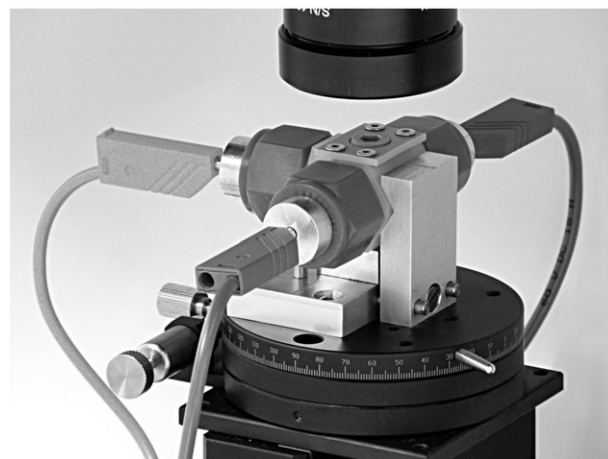


Fig. 2. Photograph of the developed cell design.

electrode potentials of anode and cathode and the cell voltage during a cycle with a C-rate of 0.5 C (1.5 mA cm^{-2}). Data are presented as a function of the specific capacity which is calculated from the consumed charge and the mass of LCO in the cathode. The characteristic features of LCO [15] and graphite [16] are clearly observed. Thus, parasitic reactions can be excluded.

Fig. 4 shows a collage of a schematic view and a light microscopic picture of the battery stack as well as the temporal evolution of thermograms during charging at 5.0 C. A significant increase in temperature is clearly observed after 15 s of charging. This behavior progresses up to 120 s when the current is interrupted to analyze thermal relaxation.

Different temperatures of the individual components within the single battery stack are observed. This indicates differences in the local heat generation rates. Obviously, the cathode dominates heat generation and a stationary temperature gradient over the cross section of the single battery stack is formed.

Assuming negligible heat of mixing as well as no phase changes the local heat generation is calculated according to [17]:

$$\dot{q} = j \left(\sum \eta - \frac{T \Delta S}{zF} \right) \quad (1)$$

where j is the applied current density, $\sum \eta$ is the overpotential and T , z , F , ΔS are the temperature, the valance, Faraday's constant and the entropy change due to the electrode reaction. The overpotential and the entropy change can distinctly differ between the individual

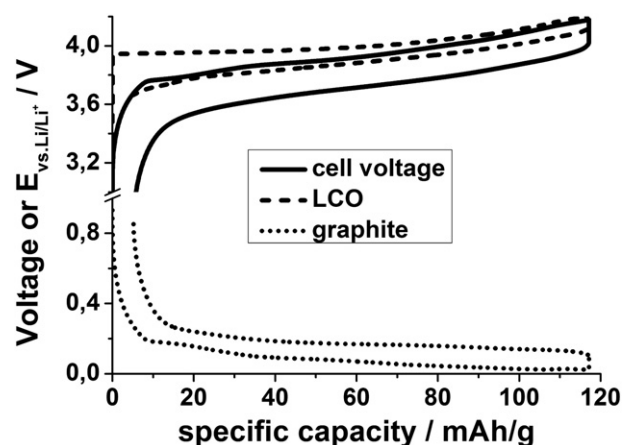


Fig. 3. Charge–discharge curves obtained by using the developed cell design.

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