



In-line monitoring of Li-ion battery electrode porosity and areal loading using active thermal scanning - modeling and initial experiment



Przemyslaw Rupnowski ^{a,*}, Michael Ulsh ^a, Bhushan Sopori ^a, Brian G. Green ^a, David L. Wood III ^b, Jianlin Li ^b, Yangping Sheng ^b

^a National Renewable Energy Laboratory, Chemistry & Nanoscience Department, 15013 Denver West Parkway, Golden, CO 80401, United States

^b Oak Ridge National Laboratory, National Transportation Research Center, Energy & Transportation Science Division, 2370 Cherahala Blvd., Knoxville, TN 37932, United States

HIGHLIGHTS

- Active thermal scanning is investigated as in-line method to monitor porosity (or areal loading).
- Thermal response of an electrode is related to coating porosity (or areal loading) and thickness.
- Thermal model that predicts response of electrode is successfully developed.

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ABSTRACT

This work focuses on a new technique called active thermal scanning for in-line monitoring of porosity and areal loading of Li-ion battery electrodes. In this technique a moving battery electrode is subjected to thermal excitation and the induced temperature rise is monitored using an infra-red camera. Static and dynamic experiments with speeds up to 1.5 m min^{-1} are performed on both cathodes and anodes and a combined micro- and macro-scale finite element thermal model of the system is developed. It is shown experimentally and through simulations that during thermal scanning the temperature profile generated in an electrode depends on both coating porosity (or area loading) and thickness. It is concluded that by inverting this relation the porosity (or areal loading) can be determined, if thermal response and thickness are simultaneously measured.

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1. Introduction

Cost reduction and performance improvement remain as main objectives of various lithium-ion battery research projects [1]. High energy density and affordable prices are required for the batteries' widespread use in electric-drive cars. To achieve this goal, high volume advanced roll-to-roll systems are used to fabricate battery electrode components. During such production, in-line monitoring and quality control become important parts of the manufacturing system.

The main active electrochemical components of a Li-ion battery are the two electrodes, cathode and anode. Slot-die coating followed by drying and calendaring is the method most commonly used in electrode fabrication. During the coating process, a layer of slurry containing functional particles, binder and solvent is overlaid onto a metal foil [2]. After coating the wet slurry is dried in an oven where solvent evaporates resulting in a porous coating. The porosity, defined as volume fraction of empty voids, is in the range of 40–70% before calendaring. In practice the actual value of the porosity for as-coated electrodes mainly depends on solvent content in the slurry and the electrode drying conditions. Because porosity has a significant impact on battery performance, a rigorous control is required for this parameter.

After slot-die coating, the porosity is typically destructively

* Corresponding author.

E-mail address: peter.rupnowski@nrel.gov (P. Rupnowski).

measured by punching out small samples from a long roll. Calculation of porosity is then based on measurement of thickness and areal loading of the punched out sample, and solid particle density that is determined from the coating composition. In the past various other measurement techniques were also used to determine porosity of battery electrode coatings [3–6]. One such example is porosimetry [3]. In this technique a non-wetting liquid is intruded into material at high pressure and a porosity calculation is made based on Washburn's equation and the critical pressure needed to force the liquid into the material. Another technique for porosity measurement is X-ray tomography [4]. The drawback of these methods is that they are relatively slow and incompatible with the concept of in-line metrology. Therefore, a technique that could determine porosity nondestructively and at high rates for electrodes that move through a roll-to-roll production line is highly desirable.

Recently active thermography has been explored as a method for detecting defects and for monitoring physical properties of battery electrodes [5,6]. In these techniques a heat impulse (flash lamp or infrared laser) is delivered to an electrode and then a transient temperature of the electrode is recorded as a function of time using an infrared (IR) camera. It was observed that the detection of coating defects could be performed based on the analysis of the uniformity of the thermal map. It was also observed experimentally that the temperature decay rate depended on thermal properties of the coating which in turn are a function of porosity (or areal loading) and thickness. Results published in Refs. [5,6] showed that the thermal response can be correlated to physical properties of the electrode; however, the demonstration was only performed for a stationary system and there was no verification that it could also work when a battery electrode is in motion, i.e. during production.

In this paper we investigate a new non-destructive technique called active thermal scanning. The method is compatible with in-line monitoring on a roll-to-roll system because during the measurement a battery electrode moves at a constant speed. Electrodes are excited by absorption of visible or IR radiation, and the resulting thermal response is monitored with an IR camera. The technique is investigated in two ways. First, experimental data are gathered for as-coated (uncalendered) electrodes. Then a two-scale model is developed and tuned to predict the response of the thermal scanner. Using these simulations and experimental results, performed for both cathodes and anodes, we evaluate the sensitivity of the thermal response to the coating's porosity and thickness.

2. Experimental

The main components of a thermal scanner are depicted schematically in Fig. 1a. A moving battery electrode is subjected to a stationary, linear heat source. In our experiments, the motion is realized by a single-axis positioner or an industrial roll-to-roll system, depending on the intended speed. The heat source produces a beam of radiation that spans across the width of the electrode in the direction perpendicular to the motion. Due to the radiation exposure, the temperature of the battery electrode locally increases. As the electrode moves away from the heat source it loses heat through convection and radiation, and the temperature returns back to room temperature. The generated temperature rise in the electrode is continuously monitored by an IR sensor.

2.1. Specimens of battery electrodes and their composition

To evaluate the response of Li-ion battery electrodes to the described active thermal scanning, four specimens - two cathodes and two anodes - are prepared, tested and modeled. The values of

electrode coating areal loading and thickness for the specimens are given in Table 1. The porosity of electrode coating was calculated based on the loading, thickness and, so-called, solid-particle density using the formula given in Ref. [6]. Both the solid-particle density and electrode porosities are also listed in Table 1. It can be noticed that within each type of electrode both thickness and porosity are significantly different.

The compositions of the investigated battery electrodes are as follows. Each electrode consists of two layers, a composite coating and a metal substrate. In the case of the cathodes, the top layer is a composite containing $\text{LiNi}_{0.5}\text{Mn}_{0.3}\text{Co}_{0.2}\text{O}_2$ (NMC532, Toda America) and carbon black (powder grade, Denka) particles held together by a polyvinylidene fluoride (PVDF, Solvay Solef® 5130) polymer binder with the weight fractions for these three materials equal to 90/5/5, respectively. The bottom layer of the cathode is a 15 μm thick aluminum foil. In the case of the anodes, the coating consists of graphite (A12, ConocoPhillips) and carbon black (C-ENERGY Super C65, Imerys Graphite & Carbon) particles and PVDF (Kureha 9300) polymer binder where the weight fractions are 92/2/6. The anode substrate is 9 μm thick copper foil. The electrode fabrication process has been reported previously [7].

Thermal conductivity, K , isobaric specific heat capacity, cp , and density, ρ , for all the constituents were collected from the literature [8–11] and are listed in Table 2. It should be noted that properties for the NMC532 particles were not explicitly available and the assumed values in Table 2 correspond to a simple average for nickel, manganese and cobalt oxides.

2.2. Stationary electrodes subjected to switching heat source

Initially low heat and zero-speed tests were carried out on stationary specimens that were approximately 300 mm long. The heat input was provided by a fiber optic light line (Volpi Lightline DC-250/LENS) connected to a white light LED source (Volpi IntraLED 2020) with electrical power input of 16 W. The width of the heating beam was about 2 mm. The heat source was switched on until a steady state temperature was established. Then the heat source was turned off and the temperature decay was monitored as a function of time. A microbolometer IR camera (Jenoptik VariocAM high resolution) with sensitivity from 7.5 to 14 μm was used to obtain the transient response of the electrode.

2.3. Electrode at low speed subjected to constant low power heat source

In the second experiment the same setup as before is used with addition of a single axis positioner to introduce a motion. An aluminum frame is attached to the positioner and an electrode sample is fastened to the frame along two edges. Because of the relatively low heating power of the LED source, the samples were moved at a low speed of 0.15 m min^{-1} . Due to the limited length of the electrode and because of the contact with the metal frame, the temperature distribution is unstable at the beginning and end of a scan. Therefore, care was taken to focus on the middle section of a scan where a steady-state temperature profile is well established.

2.4. Roll with electrodes at higher speeds subjected to constant higher power heat source

In the third experiment the same electrode samples are spliced together and inserted into a roll of 75 μm -thick polyester polymer web. The entire roll is run through an industrial-style roll-to-roll system and a custom-built, linear radiant heater with higher power output is employed. The heater is comprised of a 12.7 mm diameter heating rod with surface modified to be of high emissivity, and an

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