



# A facile electrode preparation method for accurate electrochemical measurements of double-side-coated electrode from commercial Li-ion batteries

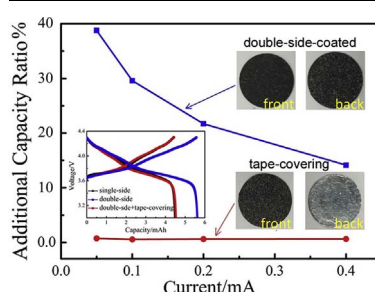
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## HIGHLIGHTS

- A facile electrode preparation method has been developed.
- Double-side-coated electrode can be directly used in simulated coin-cell.
- Accurately capacity measurement can be achieved with an error of 0.5%.
- Quantitative EIS measurement can be performed with double-side-coated electrode.

## GRAPHICAL ABSTRACT



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## ABSTRACT

The post mortem electrochemical analysis, including charge-discharge and electrochemical impedance spectroscopy (EIS) measurements, are critical steps for revealing the failure mechanisms of commercial lithium-ion batteries (LIBs). These post measurements usually require the reassembling of coin-cell with electrode which is often double-side-coated in commercial LIBs. It is difficult to use such double-side-coated electrode to perform accurate electrochemical measurements because the back side of the electrode is coated with active materials, rather than single-side-coated electrode that is often used in coin-cell measurements. In this study, we report a facile tape-covering sample preparation method, which can effectively suppress the influence of back side of the double-side-coated electrodes on capacity and EIS measurements in coin-cells. By tape-covering the unwanted side, the areal capacity of the desired investigated side of the electrode has been accurately measured with an experimental error of about 0.5% at various current densities, and accurate EIS measurements and analysis have been conducted as well.

## 1. Introduction

Lithium-ion batteries (LIBs) become an increasing concern with their widely used in consumer electronic devices, electric vehicles and electrical energy storage systems, owing to their high energy density, long cycle life, low maintenance, and environmental friendliness [1,2].

A persistent problem limiting the use of LIBs is the degradation of performance over the lifetime of the battery. For instance, cell capacity fade and power fade are the most important aging effects [3,4]. The comprehensive knowledge and appropriate model of the degradation mechanism of LIBs plays a crucial role in diagnosing and developing the batteries [5–7].

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Several techniques, such as the electrochemical analysis techniques, are commonly used to identify and quantify the effects of degradation. The common measurements for post mortem analysis include charge-discharge [8–10] and electrochemical impedance spectroscopy (EIS) measurements [11–13]. The capacity change, the incremental capacity analysis and differential voltage analysis according to the charge-discharge test of the cathode and anode are effective means for the diagnosis of cell capacity fade [14]. The accurate measurement of the areal capacity of the electrode is a direct method to quantify the origins of the degradation (cathode, anode and lithium inventory). Another widely used experimental technique, EIS was used to investigate the individual reaction process and its dynamic change occurred in the cell. By analyzing EIS data of full coin-cells and symmetric coin-cells at different frequency regions, the degradation modes could be inferred [15].

The post mortem electrochemical analysis of the LIBs is started with the appropriate sampling and preparation of the electrode samples for electrochemical measurements with reassembled simulating cells (such as coin-cell). As most commercial LIBs assembled with double-side-coated (abbreviated with DS) electrodes, the back side of the electrode poses notable influence on accurate electrochemical measurements with coin-cells. A conventional way is to scratch the active materials off from one side of DS electrode, which is time-consuming and often causes damages on the surface of the electrode. Recently, some researchers have used the DS electrodes directly in coin-cells to study the effects of electrolyte additives and pointed out that the back side of the electrode (adhered to cap of coin-cell) is basically electrochemically inactive [11].

In this study, two different kinds of LIBs containing both single-side-coated (abbreviated with SS for reference use) and DS electrodes were chosen to investigate how accurate the capacity and EIS measurements are for the direct use of DS electrode in coin-cells. A facile tape-covering sample preparation method was developed, which can significantly diminish the influence of the back side of DS electrode on the accurate electrochemical measurements and enable the direct use of DS electrode in post electrochemical analysis.

## 2. Experimental

### 2.1. Sample preparation

The  $(\text{LiNi}_{0.6}\text{Co}_{0.2}\text{Mn}_{0.2}\text{O}_2 + \text{LiMn}_2\text{O}_4)/\text{graphite}$  pouch-type cell (marked as cell 1) and  $\text{LiNi}_{0.6}\text{Co}_{0.2}\text{Mn}_{0.2}\text{O}_2/\text{graphite}$  pouch-type cell (marked as cell 2) were chosen in this study because both cells have SS cathode and anode electrodes which can be used as reference for determining the areal capacity. Both cells were provided by Beijing WeLion New Energy Technology Co., LTD with the capacity of 30Ah and 29Ah respectively. The cathode electrode of cell 1 was composed of  $\text{LiNi}_{0.6}\text{Co}_{0.2}\text{Mn}_{0.2}\text{O}_2$  and  $\text{LiMn}_2\text{O}_4$  with molar ratio of 7.8: 1 (information provided by Beijing WeLion New Energy Technology Co., LTD). Both cells were charged and discharged for 2 cycles between 3.0 and 4.3 V to activate and stabilize the cell performance and then disassembled with a ceramic scissor in an argon filled glove box ( $\text{H}_2\text{O}$  and  $\text{O}_2$  concentrations < 0.1 ppm). The jelly roll could be extracted and unrolled, comprising SS and DS electrodes as schematic shown in Fig. 1. Electrode discs were punched from the selected areas of SS and DS electrodes for further electrochemical measurements in coin-cells. These electrode discs were carefully washed with dimethyl carbonate (DMC) and dried overnight under vacuum inside the antechamber of the glove box. Half coin-cells, full coin-cells and symmetric coin-cells were assembled using CR2032 coin-cell for different electrochemical measurements, schematics of different coin-cell were shown in Fig. 2. Regular electrolyte with 1 M  $\text{LiPF}_6$  in ethylene carbonate and dimethyl carbonate (1:1 ratio by volume) and conventional Celgard separator were used.

In order to evaluate the influence of back-side coating on the accurate electrochemical measurements and data analysis for DS

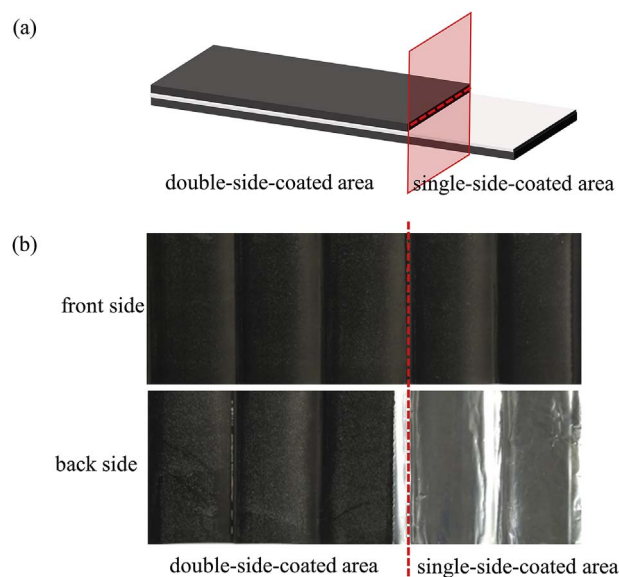


Fig. 1. (a) Schematics and (b) digital photographs of unrolled electrodes from disassembled pouch cell, including a few single-side-coated electrodes.

electrodes, five kinds of electrode discs (both cathode and anode) with different characteristics, as depicted in Fig. 2, were prepared for comparison studies. I) SS electrodes, collected from the SS area. II) DS electrodes, collected from DS area with undamaged electrode surface. III) DS electrodes, collected from DS area with damaged electrode surface on one side (might be caused during cell disassembling). IV) DS electrodes with undamaged surface, covered with Al (for cathode) or Cu (for anode) tape. V) DS electrodes with damaged surface, covered with Al or Cu tape on damaged side. The Al and Cu conducting tapes used in this study were purchased from 3M Inc (3M1170 and 3M1181, respectively) with thickness of about 0.1 mm.

### 2.2. Electrochemical measurement

Charge-discharge measurements were carried out on a LAND battery test system (Land CT2001A, China) at various current densities. In order to guarantee and demonstrate the reliability of results, paralleling tests with four coin-cells for each type of measurements were performed. Electrochemical impedance spectroscopy (EIS) measurements were conducted on an electrochemical workstation (Zahner IM6, Germany) with a.c. signals of 5 mV in a frequency range of 1M–10 m Hz at 25 °C. The electrodes for EIS measurements were collected from the batteries disassembled after resting in the fully discharged state (for cathode) and fully charged state (for anode) at 3 V for 4 h. The assembled coin-cells were standing for 24 h to achieve the equilibrium for EIS measurements.

## 3. Results and discussion

### 3.1. The capacity measurement

The graphite anodes with both SS and DS electrodes were used to evaluate how back-side coating affects the accurate determination of the areal capacity of the electrode materials. The areal capacity of SS electrode, accurately measured in coin-cell, can be used as a reference value to evaluate the accuracy of the areal capacity determined from the measurement with DS electrode. In the present work, Li/graphite half coin-cells were reassembled in CR2032 coin-cell using the graphite electrodes collected from cell 1 and cell 2 as noted. DS electrodes were directly used to measure the areal capacity of the graphite electrodes. The additional charge/discharge capacity ratio, defined as  $R_{ac} = (C_d -$

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