



Comprehensive calorimetry of the thermally-induced failure of a lithium ion battery



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HIGHLIGHTS

- Tool for measurement of energetics of thermal battery failure was developed.
- This tool was used to investigate a lithium ion battery at various states of charge.
- Time and temperature resolved data on heat production inside battery was obtained.
- This tool was combined with cone calorimetry.
- Energetics of flaming combustion of materials ejected from battery was quantified.

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ABSTRACT

A lithium ion battery (LIB) subjected to external heat may fail irreversibly. Manifestations of this failure include venting of potentially combustible gases and aerosols followed by a rapid self-heating accompanied by ejection of the battery materials. It is important to be able to quantify the dynamics and energetics of this process to ensure safety of the energy storage systems utilizing LIBs. Here we report on development of a new experimental technique for the measurement of energetics of a thermally-induced battery failure. This technique, Copper Slug Battery Calorimetry (CSBC), was employed to investigate a widely utilized LIB of 2200 mAh capacity at various states of charge (SOC). It was shown that this technique yields time and temperature resolved data on the rate of heat production inside the failing battery. The total energy generated inside the battery was found to increase with increasing SOC to the maximum value of 34.0 ± 1.8 kJ. To capture the energetics of flaming combustion of the materials ejected from the battery, CSBC was coupled with a cone calorimeter, which measures heat released in a non-premixed flame. The maximum amount of energy released by the battery through flaming combustion of ejected materials was found to be 97.5 ± 12.4 kJ.

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1. Introduction

Lithium ion batteries (LIBs) are the state-of-the-art energy storage devices employed in a wide variety of electrical and electronic systems [1]. They provide the best combination of high energy density, high efficiency and long cycle life, and exhibit no memory effect [2]. As LIBs are being deployed in an increasing range of energy demanding applications, fire safety of these devices becomes an important consideration.

LIBs typically consist of four primary components: two

electrodes (anode and cathode), electrolyte and separator [1]. In most commercial LIB cells, the anode material is graphite. The most common cathode materials are metal oxides (for example: lithium cobalt oxide or lithium manganese oxide). The electrolyte is typically a mixture of organic carbonates. The separator is a thin (<30 μm) micro-porous polymer layer [3] located between the cathode and anode to prevent internal short circuit while allowing lithium ions to pass through.

When LIBs are subjected to environmental conditions outside their intended designed envelope, they may fail irreversibly. In particular, thermal abuse may trigger rapid exothermic reactions inside an LIB. More specifically, an LIB subjected to sufficient external heat may initially vent potentially combustible gases and aerosols. This phenomenon is referred to as safety venting [2]; it

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represents an engineered mechanism that prevents internal pressure build-up and explosive rupture of the cell casing. Subsequently, the LIB may self-heat rapidly. This phenomenon is referred to as thermal runaway [1,2] because of its self-accelerating nature (e.g., increasing temperature increases the rate of chemical reactions which produce heat and further increase the temperature). The thermal runaway is frequently accompanied by ejection of a portion of electrolyte, separator and electrode materials. These two stages of thermally-induced failure can contribute to hazards associated with a potential fire occurrence. A number of fire and explosion incidents associated with LIBs have been reported, ranging from ignition of consumer electronic devices to fires in electric vehicles [1].

Considerable research effort has been directed toward measurement of onset temperatures and energetics of the thermally-induced failure of LIBs. Experimental techniques such as differential scanning calorimetry (DSC) [4–12], accelerating rate calorimetry (ARC) [9–11,13–18], VSP2 adiabatic calorimetry [12,19–21], C80 calorimetry [22–24] and oxygen consumption calorimetry [25] were utilized by various researchers to analyze the failure processes. DSC is a thermoanalytical technique that measures the heat flow associated with physical and chemical transitions in a milligram-sized sample. The sample temperature is increased linearly with time at rates that are sufficiently low (typically, 3–30 K min⁻¹) to maintain spatial uniformity of this temperature.

DSC was used to study a number of electrode and electrolyte materials as well as their combinations. Yang et al. [4] investigated graphite, the most common anode material, at various states of charge (SOC). Sharp exothermic peaks were detected at around 600 K in the samples containing more than 0.7 lithium ions per 6 carbons, and were attributed to the structural collapse of the graphitic matrix. Roth et al. [6] examined thermally-induced interactions between several binder materials (used to ensure adhesion between electrode and current collector), representative anode materials at different SOC and electrolyte. It was found that the amount of electrolyte, the surface area of the anode and its SOC have a strong impact on the exothermicity of the observed reactions. Maleki et al. [10] employed DSC to examine the thermal stability of the anode and cathode for a specific LIB chemistry, graphite/LiCoO₂. The total heats of decomposition were measured to be 697 and 407 J g⁻¹, for the anode and cathode, respectively (here, exo is positive). These heats decreased by about 60% with the removal of electrolyte.

ARC [26] measures temperature and pressure change in a sealed stainless steel or titanium container that accommodates a relatively large (about 100 cm³ in volume) sample of interest. The container is heated slowly (about 0.2 K min⁻¹). When an exothermic process inside the container is detected, the instrument increases container housing temperature to minimize the heat transfer from/to the container.

Von Sacken and co-authors [14] utilized ARC to study and compare the thermal stability of battery cells containing different anode materials. They showed that the carbon intercalation anode was superior to a lithium metal anode. Maleki et al. [10] applied this technique to measure the onset temperature of chemical reactions that force the cell into thermal runaway. This temperature was found to be close to the decomposition temperature of the unwashed cathode material.

While DSC, ARC and other aforementioned calorimetry techniques proved to be very useful in analysis of LIB failure, they possess notable limitations. For example, DSC can only be applied to the analysis of battery components. ARC has not been specifically designed for LIB failure experiments, where chemical processes inside and outside the battery casing may contribute to the energy generation and the rates of temperature rise may exceed

500 K min⁻¹. A quantitative interpretation of the ARC data on LIB failure can be difficult.

Here we propose a simple experimental technique designed specifically to measure energetics of a thermally-induced LIB failure. This technique, which we refer to as Copper Slug Battery Calorimetry (CSBC), measures the amount of energy released inside a casing of an LIB cell as it undergoes the failure process. The apparatus that was employed in this study is specific to the 18650 form factor of LIB cells (which are cylinders 18 mm in diameter and 65 mm in height). However, the technique can be readily adapted to a range of cell shapes and sizes. We also demonstrate how the CSBC can be combined with cone calorimetry [27] to enable measurement of the energy released as a result of a flaming combustion of the battery materials ejected from the cell. A comprehensive characterization of one LIB type, Panasonic CGR18650CG, is carried out to demonstrate the capabilities of this technique. The dependence of failure energetics on the battery's SOC is analyzed and discussed.

2. Experimental

2.1. Battery samples

Panasonic CGR18650CG LIB cells were examined in this study. These cells contain lithium nickel manganese cobalt oxide cathode and carbon anode; their electrolyte is composed mainly of alkyl carbonate [28]. The cells have a nominal capacity of 2200 mAh and a nominal voltage of 3.6 V. They are equipped with three safety vent ports, which are located at the positive end of the battery. The cells were stripped of plastic packaging prior to testing. The mass of the cells without the plastic packaging was found to be 42.30 ± 0.04 g. A photograph of the LIB with and without the plastic packaging is shown in Fig. 1.

An iCharger 208B battery charger controlled by a computer was used to prepare cell samples at specific SOCs. Each battery was first fully discharged to a minimum voltage of 2.5 V using a constant current of 1.0 A. Subsequently, the battery was charged to 4.2 V using the constant current/constant voltage method until the charge current fell below 0.1 A. The capacity of each cell was measured during this charging process and was found to be within 3% of the nominal capacity. Finally, the battery was discharged using a constant current of 1.0 A until the desired SOC was reached, as calculated by current integration. The chosen states of charge for this study were 100, 50, 25 and 0% SOC.



Fig. 1. Panasonic CGR18650CG LIB cell with (left) and without (right) plastic packaging.

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