Lithium-Ion Battery Thermal Runaway Electro-Thermal Triggering Method and Toxicity Analysis

H J Xie¹, J Sun², J G Li¹, T Zhou¹, S P Wei¹ and Z H Yi¹

¹Department of Chemistry Defense, Institute of NBC Defense, Beijing 102205, China

E-mail: magnsun@mail.tsinghua.edu.cn

Abstract. In high temperature condition, lithium-ion batteries have a greater risk of thermal runaway. Lithium-ion batteries may be exposed to smoke, combustion, or even explosion, which poses a greater threat to humans and the environment. In this paper, the electro-thermal triggering method is used to induce thermal runaway of the lithium-ion batteries. The most representative ternary polymer lithium-ion battery (NMC), lithium cobalt oxide battery (LCO), lithium iron phosphate battery (LFP) in the market were selected as experimental samples, all using 18650 batteries, and then the state of charge of battery samples were adjusted to 0%, 30%, 50%, 100%. The thermal runaway reaction phenomenon of lithium-ion batteries and the surface temperature of the batteries were recorded. The positive electrode samples of the batteries were characterized by X-ray diffraction and electron microscopy, and the thermal runaway reaction products were analyzed. The results show that with the increase of the state of charge, the thermal runaway reactions of the batteries are more severe. Thermal runaway reaction products contain a large amount of toxic substances, and prevention and protection are necessary.

1. Introduction

With the development of the world, a large amount of natural energy has been developed and utilized by people, which has led to an energy crisis in the world [1]. Therefore, the research of new energy sources is of great significance. It is widely believed that lithium-ion batteries will become one of the major candidate power sources for the 21st century [2]. Lithium-ion batteries have the advantages of large specific energy, high output voltage, long cycle life and low environmental pollution [3]. They are widely used in microelectronics, electric vehicles, photovoltaic engineering, military, space technology and other fields [4]. Lithium-ion batteries have potential safety hazards in the process of use, which affects people's safety [5]. The main obstacles and bottlenecks restricting the popularization and application of lithium-ion batteries are battery safety issues [6].

For the safety and reliability of lithium-ion batteries, relevant organizations and institutions at home and abroad have established test standards for lithium-ion batteries to meet actual application needs[7]. The test standard provides a detailed evaluation of battery safety and reliability under extreme conditions such as short circuit, overcharge and over discharge, collision, and smoke [8].

In the preliminary work of this group [9], the mechanism of thermal runaway reaction of lithium-ion battery was summarized. Our group used flame triggering method to conduct thermal runaway experiments on two types of commercial lithium-ion batteries (18650 lithium-ion batteries and punch lithium-ion batteries). However, this method inevitably introduces impurities and adversely affects the test results. We use the electrical heating trigger to replace the original method, so that we can reduce
the influence of impurities and achieve the purpose of temperature control.
In this paper, we use the 18650 lithium-ion batteries as the research object to explore the electric heating trigger method of lithium-ion batteries [10]. We used the electrical heating trigger on 18650 lithium-ion batteries with different SOCs, and analyze the surface temperature of the lithium-ion batteries, the temperature change of the leaking flue gas and the reaction leakage gas during the triggering process [11].

2. Experimental section

2.1. Selection of lithium-ion batteries types
The experimental batteries (18650 LIB) were divided into three types according to the cathode material: Ternary Polymer Lithium Ion Battery (NMC), lithium cobalt oxide battery (LCO), lithium iron phosphate battery (LFP). Four sets of samples were prepared for each kinds of battery, and the battery state was adjusted to 0%, 30%, 50%, 100% using a battery integrated tester [12]. The performance parameters of various LIBs are listed in Table 1.

| LIB Types | Nominal Capacity/mA·h | Nominal Voltage/V | Over Charge Protection Voltage/V | Over Discharge Release Voltage/V |
|-----------|------------------------|-------------------|----------------------------------|----------------------------------|
| NMC       | 2200                   | 3.70              | 4.20                             | 2.75                             |
| LCO       | 2600                   | 3.70              | 4.20                             | 2.75                             |
| LFP       | 1300                   | 3.20              | 3.75                             | 2.50                             |

2.2. Thermal runaway initiation experiment
The test box was a 500*500*500 mm³ stainless steel apparatus. The heating plate heats the 18650 lithium-ion battery to cause thermal runaway of the lithium-ion batteries. The temperature of electric heating trigger is 170 °C. Lithium-ion battery is fixed by wire and placed on the surface of the heating plate. Thermocouple detects the temperature of the electric heating plate, the surface of lithium ion batteries, and the gas environment. The temperature measurement points were shown in Figure 1. High frame camera was used to record experimental phenomenon.

![Figure 1](image.png)

**Figure 1.** Thermal runaway electrical heating trigger for Li-ion batteries
a. The heating plate b. The surface of lithium ion batteries c. The gas environment

2.3 Detection of thermal runaway reaction products
In this paper, GC-Mass (GC–MS Hapsite) is used as a thermal runaway reaction gas collection and analysis device for lithium ion batteries, including toxic industrial raw materials (Rapid monitoring and analysis of substances such as TIM) and toxic industrial chemicals (TIC). After the gas environment was lowered to the room temperature, the GC-Mass was used to collect and analyze the thermal runaway reaction gas.

3. Results and discussion

Lithium-ion battery is thermal runaway triggered at 170 ℃. The purpose of this experiment is to record the experimental phenomenon and the temperature of measurement points, and sample the reaction gas to analyze the damage of the thermal runaway reaction to the human body and the environment [13].

3.1 Experimental phenomena and temperature analysis

In the previous step, the lithium-ion batteries were charged to different SOCs (0%, 30%, 50%, 100%). The Lithium-ion batteries were triggered by thermal runaway under the same experimental condition. The temperatures of cell surfaces as well as experimental phenomena were recorded by laboratory equipment [14].

![Figure 2. Thermal runaway temperature curve of NMC Li-ion batteries](image)

As shown in Figure 2, when the temperature of the heating plate rose to the set temperature (170 ℃), the other three batteries were rising rapidly except for the battery with 0% SOC. The batteries with 30% and 50% SOCs had very similar curves. Both batteries had electrolyte leakage with a small amount of smoke in 5 minutes. After eight minutes, a lot of smoke popped up in the reaction box. The experimental phenomenon is shown in Figure 3. The battery with 100% SOC exploded in 8 minutes with a flare, as shown in Figure 4.
Figure 3. The Thermal runaway reaction phenomenon of NMC Li-ion batteries
a. 30% SOC b. 50% SOC

Figure 4. The Thermal runaway reaction phenomenon of NMC Li-ion battery with 100 % SOC

Figure 5. Thermal runaway temperature curve of LCO Li-ion batteries
As shown in Figure 5. The temperature of battery with 50% SOC rose rapidly at the initial stage and reached its peak in 9 minutes. The temperature of the battery with 100% SOC also reached its peak at 9 minutes and exploded with dazzling flares. The Battery internal material burns violently. The experimental phenomenon is shown in Figure 6.

![Image](image_url)

**Figure 6.** The Thermal runaway reaction phenomenon of LCO Li-ion battery with 100 % SOC

![Graph](graph_url)

**Figure 7.** Thermal runaway temperature curve of LFP Li-ion batteries

Compared to the previous two types of batteries, the temperature of LFP Li-ion batteries rose rapidly at an early stage. It’s shown in Figure 7. The other three batteries exploded except the battery with 30% SOC. In the current battery transportation process, the battery is usually adjusted to 30-70%. ICAO (International Civil Aviation Organization) requires that the upper limit of battery transportation power is not higher than 30% SOC. Generally, lithium-ion batteries at 30% SOC are considered to have better Safety performance.

3.2. Thermal runaway reaction gas analysis

The GC-Mass was used to collect and analyze the thermal runaway reaction gas. Six classification levels including I (very toxic), II (highly toxic), III (toxic), IV (harmful) and V (low toxic) and VI (Few Toxic) level are defined according to China National Standards including GB5044-1985 Classification of health hazard levels from occupational exposure to toxic substances and GBZ230-2010 Classification for hazards of occupational exposure to toxicant [9]. Three of the most dangerous levels (I, II. III) of poisons were selected. The analysis results were shown in the Table 2. The thermal runaway reaction product of LCO Li-ion battery had more kinds of poisons. Among all types of batteries, 100% SOC batteries had more types of poisons. The 100% SOC is the most dangerous state of charge terms of toxicity and
hazards. Compared with the previous work of this group, more kinds of poisons are detected, such as Ethyl thiocyanate, 1,1-Dimethyldrazine, Ethyl thiocyanate.

2-Propenal is very toxic with highly irritating and tear-prone. It is located in the list of carcinogens published by the World Health Organization's International Agency for Research on Cancer [15]. After the SEI film is destroyed, the lithium embedded in the negative electrode reacts directly with the electrolyte solvent to form a stable SEI film and combustible hydrocarbons [16]. The generated propylene is oxidized to 2-Propenal at high temperature.

\[
2 \text{Li} + \text{C}_4\text{H}_6\text{O}_3(\text{PC}) \rightarrow \text{Li}_2\text{CO}_3 + \text{C}_3\text{H}_6
\]

\[
\text{C}_3\text{H}_6 + \text{O}_2 \rightarrow \text{CH}_2=\text{CHCHO} + \text{H}_2\text{O}
\]

Propylene oxide and 1,2-epoxybutane are irritating to the respiratory tract. These substances are mainly converted from organic substances in the electrolyte [17].

Table 2. Test result of thermal runaway products of Li-ion batteries under different SOC by GC/MS

| NO. | Compound                        | CAS     | Toxicity classification | NMC | LCO | LFP |
|-----|---------------------------------|---------|-------------------------|-----|-----|-----|
| 1   | 2-Propenal (C3H4O)              | 107-02-8| I                       | 30% | 100%|     |
| 2   | Dimethyl hydrazine (C2H8N2)     | 540-73-8| I                       |     | 100%|     |
| 3   | Methyl vinyl ketone (C4H6O)     | 78-94-4 | I                       | 100%| 100%|     |
| 4   | Propanedinitrile (C3H2N)        | 09-77-3 | I                       | 100%|     |     |
| 5   | Propanenitrile (C3H5N)          | 107-12-0| I                       | 100%|     |     |
| 6   | Ethyl thiocyanate (C3H5NS)      | 542-90-5| I                       |     |     | 30% |
| 7   | 1,3-cyclopentadiene (C5H6)      | 542-92-7| II                      | 100%| 50% | 100%|
| 8   | n-Butylamine (C4H11N)           | 109-73-9| II                      | 50% |     |     |
| 9   | Crotonaldehyde (C4H6O)          | 4170-30-3| II                      | 50% |     |     |
| 10  | Allyl alcohol (C3H6O)           | 107-18-6| II                      | 50% |     |     |
| 11  | Acrylic acid (C3H4O2)           | 79-10-7 | II                      |    | 30% |     |
| 12  | 2-Furaldehyde (C5H4O2)         | 98-01-1 | II                      |    | 50% |     |
| 13  | 1,1-Dimethyldrazine (C2H8N2)   | 57-14-7 | II                      |    | 30% |     |
| 14  | Naphthalene (C10H8)            | 91-20-3 | II                      |    | 50% | 100%|
| 15  | 1,2-epoxybutane (C4H8O)        | 106-88-7| II                      |    | 50% |     |
| 16  | Propylene oxide (C3H6O)        | 75-56-9 | II                      |    |     | 30% |
3.3. XRD research on the structural transformation of materials after thermal runaway reaction

![XRD results of NMC thermal runaway reaction cathode material with different SOCs](image)

**Figure 8.** XRD results of NMC thermal runaway reaction cathode material with different SOCs

![XRD results of LCO thermal runaway reaction cathode material with different SOCs](image)

**Figure 9.** XRD results of LCO thermal runaway reaction cathode material with different SOCs

![XRD results of LFP thermal runaway reaction cathode material with different SOCs](image)

**Figure 10.** XRD results of LFP thermal runaway reaction cathode material with different SOCs
After the end of the thermal runaway experiment, the positive electrode material of the battery was collected, and the positive electrode material sample was subjected to X-ray diffraction detection and analysis [18].

It’s shown in Figure 8. In the XRD spectrum of the blank sample, two main peaks (003) and (104) appear at 2θ=18° and 2θ=45°, respectively, and the split peak at (006)/(102) was weak. There were obvious split peaks at (018)/(110), indicating that the three proportions of lithium-rich cathode materials form a typical layered crystal structure [19]. The battery sample with 0% SOCs retained the hexagonal layered crystal structure after the thermal runaway reaction. The battery sample with 30% SOCs was not completely split at (006)/(012) and (108)/(110), indicating that the layered structure of the material may be destroyed, which may be decomposition of the positive electrode material [20]. The reaction and the layered structure were destroyed. MnF characteristic peak existed in the battery sample with 30% SOCs, indicating that the cathode material reacts with the electrolyte after decomposition [21]. The battery sample with 100% SOCs showed a significant carbon characteristic peak at 2θ=28°. In the thermal runaway reaction, part of the carbon powder was doped into the positive electrode material through the broken diaphragm [22].

XRD analysis of the residue samples of LCO showed that the structure of the sample changed. As shown in Figure 9. The battery sample with 0% SOCs has been damaged due to high temperature for a long time, and the negative electrode carbon is mixed into the positive electrode, which shows a carbon characteristic peak in the XRD spectrum [23]. In the sample of 30% SOC ternary polymer lithium ion battery, the positive electrode aluminum foil was oxidized at high temperature, and the characteristic peak of Al2O3 appeared at 2θ=26°; the decomposition of the positive electrode material was observed, and the existence of CoO was found in the XRD spectrum [24]. In the sample of 50% SOC lithium ion battery, the presence of Li2O was found to indicate that Li was completely oxidized in the battery at high temperatures [25]. In the sample of 100% SOC ternary polymer lithium ion battery, Co3O4 was found to exist in CoO and Co [26]. It is indicated that after the battery separator is melted, the cathode material undergoes a decomposition reaction to release oxygen.

\[
\text{Li}_{x}\text{CoO}_2 \rightarrow x \text{LiCoO}_2 + \frac{1}{3}(1-x)\text{Co}_3\text{O}_4 + \frac{1}{3}(1-x)\text{O}_2
\]

\[
\text{CO}_3\text{O}_4 \rightarrow 3 \text{CoO} + 0.5 \text{O}_2
\]

\[
\text{CoO} \rightarrow \text{Co} + 0.5 \text{O}_2
\]

XRD analysis was performed on the residue of LFP. It’s shown in Figure 10. The characteristic diffraction peak of the XRD pattern of the blank sample is consistent with the peak position and relative intensity of the standard diffraction peak, indicating that the positive electrode sheet material of the blank sample is an ordered olivine-type structure [27]. In the battery sample with 0% SOC, compared with the blank sample, a significant carbon characteristic peak appeared at 2θ=28°, indicating that the battery separator was damaged and the anode material was doped into the positive electrode [28]. The impurity peaks of LiF and Fe2O3 were also found therein, indicating that lithium embedded in the negative electrode reacts with the electrolyte to form LiPF6, and then LiPF6 decomposes to form LiF, and passes through the broken separator to enter the positive electrode [29]. The lattice of the positive electrode material changes at a high temperature, which is expressed as an impurity peak of Fe2O3.

\[
2 \text{Li} + 2 \text{EC} \rightarrow \text{Li}_2\text{O} - (\text{CH}_2)_{x}\text{O} - \text{Li} + 2 \text{CO}_2
\]

\[
\text{LiPF}_6 \rightarrow \text{LiF} + \text{PF}_5
\]

The XRD spectrum of the battery sample with 30% SOC was similar to the battery sample with 0% SOC. In the XRD spectrum of the battery sample with 50% SOC, the presence of Li2CO3 was found, indicating that Li reacted with organic solvent in the anode material to form Li2CO3 [30].

\[
2 \text{Li} + 3\text{C}_x\text{H}_y\text{O}_z(\text{EC}) \rightarrow \text{Li}_2\text{CO}_3 + \text{C}_2\text{H}_4
\]

\[
2 \text{Li} + 3\text{C}_x\text{H}_y\text{O}_z(\text{PC}) \rightarrow \text{Li}_2\text{CO}_3 + \text{C}_3\text{H}_6
\]

\[
2 \text{Li} + 3\text{C}_x\text{H}_y\text{O}_z(\text{DMC}) \rightarrow \text{Li}_2\text{CO}_3 + \text{C}_3\text{H}_6
\]

In the XRD spectrum of the battery sample with 50% SOC, the carbon characteristic peak intensity is large, and the Li material has been oxidized to Li2O [31].
3.4. Electron microscope research on the structural transformation of materials after thermal runaway reaction

**Figure 11.** Electron microscope results of NMC thermal runaway reaction cathode material with 0% SOC

(a): TEM; (b): SEM

**Figure 12.** Electron microscope results of NMC thermal runaway reaction cathode material with 30% SOC

(a): TEM; (b): SEM
Figure 13. Electron microscope results of NMC thermal runaway reaction cathode material with 50% SOC

(a) TEM; (b) SEM

Figure 14. Electron microscope results of NMC thermal runaway reaction cathode material with 100% SOC

(a) TEM; (b) SEM

It can be seen from Figure 11 that after the thermal runaway reaction of NMC at 0% SOC, the positive electrode material still maintains a good tight layered structure, and the particles are closely packed inside the structure [32]. It is speculated that the sample still retains the hexagonal layered crystal structure after the NMC thermal runaway reaction at 0% SOC. It’s shown in Figure 12. In the sample after 30% SOC thermal uncontrolled reaction, the SEI film was destroyed, the layered structure was destroyed and tearing occurred, and the particles were exposed to the outside of the layered structure [33]. As shown in Figure 13, in the sample after 50% SOC thermal uncontrolled reaction, the layered structure of the material was destroyed, the particles were scattered outside the original layered structure, and adhesion occurred. The positive electrode material reacted at high temperatures [34]. Decomposed, and carbon particles of the negative electrode material are doped into the positive electrode through the damaged diaphragm. It’s shown in Figure 14. In the sample after the thermal runaway reaction of NMC
at 100% SOC, the structure of the positive electrode sample was completely destroyed, and the particles were scattered outside the original layered structure, and the adhesion phenomenon occurred, showing fragmentation. Particles cannot be found and external impurities are doped into the interior of the structure [35].

Figure 15. Electron microscope results of LCO thermal runaway reaction cathode material with 0% SOC
(a) : TEM; (b): SEM

Figure 16. Electron microscope results of LCO thermal runaway reaction cathode material with 30% SOC
(a) : TEM; (b): SEM
Figure 17. Electron microscope results of LCO thermal runaway reaction cathode material with 50% SOC

(a):TEM; (b):SEM

Figure 18. Electron microscope results of LCO thermal runaway reaction cathode material with 100% SOC

(a):TEM; (b):SEM

As can be seen from Figure 15, a tear separation of a part of the lithium cobaltate layer structure occurred in the sample, the porous structure collapsed, and a small amount of carbon particles were mixed into the positive electrode due to the damage of the separator [36]. It can be seen in Figure 16 that the particles are still inside the gel, but the structure has been destroyed, and some of the current collector fragments may be doped into the positive electrode material [37]. In Figure 17, the crystal structure of the sample was completely destroyed, and the particles were detached in the gel, and the positive electrode material exhibited a floc structure. It can be seen in Figure 18 that the particles are destroyed and may have reacted, and impurities are mixed with the material [38].
**Figure 19.** Electron microscope results of LFP thermal runaway reaction cathode material with 0% SOC
(a): TEM; (b): SEM

**Figure 20.** Electron microscope results of LFP thermal runaway reaction cathode material with 30% SOC
(a): TEM; (b): SEM

**Figure 21.** Electron microscope results of LFP thermal runaway reaction cathode material with 50% SOC
(a): TEM; (b): SEM
Figure 22. Electron microscope results of LFP thermal runaway reaction cathode material with 100% SOC

(a):TEM; (b):SEM

As shown in Figure 19, the positive electrode material still maintains a good tight layer structure. It is speculated that the sample still retains a hexagonal layered crystal structure after the thermal runaway reaction of LFP at 0% SOC, and the particles are evenly loaded inside the gel [39]. The sample shown in Figure 20 is similar in appearance to the previous set of samples. As shown in Figure 21, the layered structure of the material is destroyed, the particles are scattered outside the original layered structure, and adhesion occurs, the positive electrode material is decomposed at a high temperature, and the carbon material of the negative electrode material is damaged [40]. The separator is doped into the positive electrode. As shown in Figure 22, the material structure is in a fragment shape, and the pore structure is destroyed [41].

4. Conclusion

For ternary polymer lithium-ion batteries and lithium cobalt oxide batteries, with the increase of SOC of lithium-ion batteries, the thermal runaway reaction of batteries is more intense, the temperature of thermal runaway is reduced, and the structural damage of electrode materials is strong; under high SOC Lithium-ion batteries produce more smoke; in 100% SOC battery samples, the battery exploded. For the lithium iron phosphate battery, except for the battery sample at 30% SOC, the other groups of samples exploded. Under high temperature conditions, the internal diaphragm of the battery has been destroyed, which will cause a short circuit inside the battery and aggravate the thermal runaway reaction. The structure of the positive electrode material is destroyed in the thermal runaway reaction, and the particles are exposed and scattered outside the structure and adhered.

The HAPSITE GC-MS was used to qualitatively detect and analyze the thermal runaway reaction gas products of the battery. Each type of lithium ion battery detected dozens of toxic products, and the acryl aldehyde and butenone were detected according to national standards. Six highly toxic substances such as dinitrile, propionitrile, ethyl thiocyanate and 1,2-dimethylhydrazine were detected. People need to take the necessary preventive and protective measures to reduce the impact of toxic substances.

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