

## ARTICLE

# Effect of Amine Additives on Thermal Runaway Inhibition of SiC||NCM811 Batteries

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## Abstract

The high energy density of NCM batteries with high nickel content is a key advantage in replacing fossil fuels and promoting clean energy development, at the same time, is also a fundamental cause of serious safety hazards in batteries. Primary and secondary amines can lead to ring-opening polymerization of common ethylene carbonate electrolytes, resulting in an isolation layer between the cathode and the anode, and improving the thermal safety of the battery. In this work, the safety of batteries is considered both at the material level and at the cell level, based on the chemical reactions between amines and the battery components. At the material level, the effect of the presence or absence of amine additives on the thermal stability of the different components of the lithium-ion battery was tested by differential scanning calorimetry. At the cell level, the safety of the whole battery with and without additives was tested by using accelerating rate calorimeter to extract thermal runaway (TR) characteristic temperatures. The addition of the amine resulted in an earlier onset of some of the chemical reactions between the battery components, as well as a significant reduction in total heat release and a decrease in the maximum temperature rise rate, such that TR, was effectively suppressed.

**Keywords:** Lithium-ion batteries; Thermal runaway inhibition; Amine additive; Electrolyte; Polymerization reaction

## 1. Introduction

As well known, environmental and energy issues are significant issues of the 21st century. In order to promote the transformation of energy structure in the world, lithium-ion batteries (LIBs) and electric vehicles have come into being. High-nickel NCM batteries have high energy density and a wide range of applications, but poor thermal stability limits the development of the NCM batteries [1]. Thermal runaway (TR) of LIBs may cause serious safety hazards, where reactions between electrodes and internal short circuits serve TR [2]. According to the study on the reaction sequence of the TR process of LIBs [3–6] this process is divided into three stages, which exhibit exothermic characteristics

ranging from slow to violent. It is worth noting that the electrolyte and the anode participate in all stages of the TR process. Silicon-based anode batteries are a new direction for improving the electrical performance of lithium-ion batteries [7,8]. Meanwhile, the silicon-based anodes also show a subtly different TR mechanism, compared to the conventional graphite anodes. After changing the raw material of the anode, the exotherm of the anode with the other components seems to be a little more intense. This is shown by the fact that when the cell reaches a self-generated thermal temperature, e.g., 85 °C ±, the fully charged anode comes into direct contact with the electrolyte as the SEI film ruptures and undergoes a chemical reaction of SEI film dissolution-regeneration. This

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reaction produces reducing gases and exothermic heat. The cathode undergoes an exothermic chemical reaction with the electrolyte, which together with the anode-electrolyte reaction heats up the cell to the TR trigger temperature. The cathode starts a phase change at  $180\text{ }^{\circ}\text{C} \pm$  to produce oxygen and a redox reaction with the electrolyte, and a redox reaction with other reducing gases. After the separator has been heat crumpled or melted, a large internal short circuit between the cathode and anode heats the cell up to the maximum TR temperature [9].

Shi et al. [10] proposed a suppression method to cope with TR of LIBs due to mechanical abuse by Dibenzylamine (DBA) in 2016. An agent package containing DBA was placed inside the battery and a puncture experiment was used to simulate the TR triggered by mechanical abuse. It is found that there is a reduction of the maximum temperature rise during TR by 50% and a decrease of the maximum temperature rise rate by 30% by adding 4% mass fraction of DBA to the battery cell. The corresponding mechanism is explained that DBA has the properties of a Lewis base and inhibits the solvation of  $\text{LiPF}_6$  by a reaction with the electrolyte, and changes the electrolyte properties, so that inhibits the TR. Subsequent studies have shown that primary and secondary amines can lead to a ring-opening polymerization reaction with the common ethylene carbonate-containing electrolyte [11]. This work provides a direction to inhibit TR in LIBs.

Based on this study, our work introduces some amine additives as TR reaction inhibiting reagents in  $\text{SiC}||\text{NCM811}$  batteries, and explores a method to inhibit TR in  $\text{SiC}||\text{NCM811}$  batteries by testing the effects of the additives on the heat release characteristics and reaction sequence of the battery materials.

## 2. Experimental

We used a  $\text{SiC}||\text{NCM811}$  pouch battery with a capacity of 5 Ah from RiseSun MGL New Energy Technology Co. The charged battery was disassembled in an argon filled glove box and soaked with dimethyl carbonate (DMC). Meanwhile, after 24 h the electrode material was scraped using a scalpel and made into a powder for collection to be subsequently thermally analyzed for experiments.

### 2.1. Thermal stability testing

A Differential Scanning Calorimetry (DSC) was used to test the thermal stability of battery materials containing additives to investigate the

influence of amine additives on the thermal properties and reaction sequence of the battery materials.

We also prepared the samples to be tested in a glove box filled with argon gas. The DSC matching test aluminum crucible, consisting of an aluminum crucible and an aluminum lid, was first weighed to use a microbalance (Secura 225D, Sartorius). The total masses of the crucible and lid for  $W_{\text{pan} + \text{lid}}$  were recorded. To prepare the sample, a Sample spoon (Netzsch) was used to add the solid powder. A pipette was used to add liquid reagents. The mass of the powdered materials added was determined via using a microbalance and the powder was added to the crucible using the spoon, with pressing the crucible to seal it using the crucible press. The total mass of the crucible with the lid plus the materials,  $W_{\text{all}}$ , was recorded, as well as the  $W_{\text{mat}}$  obtained by  $W_{\text{all}}$  minus  $W_{\text{pan} + \text{lid}}$ , which is the mass of the added materials.

By varying the ratio of the active material in the cell, we prepared samples with a mixture of different components, as shown in Table 1. Simultaneously, such samples are defined as the control samples. The  $\text{SiC}||\text{NCM811}$  pouch cell used in this work has a mass ratio of active material of anode: cathode: electrolyte = 1.25 : 2 : 1.

Based on the above sample preparation, a certain amount of amine additives was added to the battery material test samples and two types of test samples were formed, as shown in Table 2. The first type of sample is an all component sample (Ca/An/Ely/Add), which is used to simulate the effect of amine additives on the TR process of the complete battery. The second type of sample is a partial component sample (Ca/Ely/Add, An/Ely/Add), which is used to investigate the effect of amine additives on the key reactions of the TR process.

### 2.2. Thermal safety testing

We used an Accelerating Rate Calorimeter (ARC) to test the battery for step-by-step self-heating TR, referred to as the ARC test. This instrument, Accelerating Rate Calorimeter, could simulate the thermal safety of a sample in a near-

Table 1. Information on samples tested for thermal safety of battery materials.

Sample category	Sample component	Abbreviation
All components sample	Cathode/Anode/Electrolyte	Ca/An/Ely
Partial components sample	Cathode/Electrolyte	Ca/Ely
	Anode/Electrolyte	An/Ely
	Cathode/Anode	Ca/An

Table 2. Information on samples tested for thermal safety of materials with amine additives.

Sample category	Sample component	Abbreviation
All components sample	Cathode/Anode /Electrolyte/Additives	Ca/An/Ely/Add
Partial components sample	Cathode/Electrolyte /Additives Anode/Electrolyte /Additives	Ca/Ely/Add An/Ely/Add

adiabatic environment, and record time-temperature-pressure data of chemical reactions under adiabatic conditions in real time [12]. This could find three important characteristic temperatures during TR:  $T_1$  – the onset temperature of self-heating;  $T_2$  – the TR temperature; and  $T_3$  – the highest temperature. In this work, an ARC (EV<sup>+</sup>, Thermal Hazard Technology) from THT (UK) was used.

Feng et al. [13] reported that there is a huge temperature difference between the inside of the battery and the surface of the battery, when TR occurs, even performing a difference of several hundred degrees Celsius between the two. This difference would lead to a different result for the extraction of the TR characteristic parameters and the analysis of the thermal safety of the battery. Xu et al. [14] reported that placing thermocouples inside the battery to obtain the internal temperature could reproduce the internal TR process of the battery more accurately. Based on the above study, we placed thermocouples on the battery to collect the internal temperature data of the battery and use the data to analyze the result of the TR characteristics of the battery.

For ARC testing, in addition to the thermocouple placed into the cell, an ARC bomb thermocouple was attached to the geometric centre of the battery surface, while a K-type thermocouple was placed on the surface to collect temperature data. This can be compared with the bomb thermocouple data to ensure that the bomb thermocouple data is accurate. In this work, a data acquisition device (LR8450, HIOKI) from HIOKI Japan was used to collect the temperature and voltage data from the battery.

### 3. Result and discussion

#### 3.1. DSC testing of battery materials

The results of DSC testing of the individual component of the cell are shown in Fig. 1. The cathode material and the anode material exhibit exothermic properties. Specifically, the cathode material has two distinct exothermic peaks, and the first exothermic peak is considered to be the

evolution of the cathode material from a laminar to a spinel I structure, as well as the second exothermic peak being the evolution from a spinel I to a spinel II structure. The anode material exhibits an exothermic phase starting at about 275 °C. The heat comes from the reaction between the lithium in the anode and the binder. The electrolyte and separator have significant heat absorption characteristics in the DSC test. The main solvents of the electrolyte used in the experimental cell are Ethyl Methyl Carbonate (EMC), Diethyl Carbonate(DEC) and Ethylene Carbonate(EC), which have boiling points of 107 °C, 126 °C and 248 °C respectively. Therefore, in the heat flow curve of the electrolyte, the first heat absorption peak (100–150 °C) comes from the evaporation of the chain carbonate solvents EMC and DEC; the second heat absorption peak comes from the evaporation of the cyclic carbonate solvent EC. The DSC test result of the separator shows that the melting process starting at 128 °C, and the complete melting and collapse of the separator at approximately 150 °C.

The DSC results for the reactions by partial components are shown in Fig. 2. In order of reaction onset temperature (Onset point), the anode/electrolyte combination is the first to start generating heat. This heat comes from the reaction between the anode and electrolyte to reform the SEI film after the decomposition of the SEI film on the anode surface, followed by cathode/anode/electrolyte, cathode/electrolyte, and anode/cathode, respectively. In this case, the cathode/electrolyte combination has approximately the same reaction start temperature and reaction end temperature as the cathode/anode combination. Comparing the heat production characteristics of the cathode in Fig. 1b, we believe that the heat production source between the cathode and electrolyte, and between the cathode and anode, is related to the oxygen production from the cathode.

In order of reaction normalized heat production (Enthalpy 50 °C–550 °C), the order is cathode/anode/electrolyte, cathode/electrolyte, anode/cathode, and anode/electrolyte in descending order. This indicates that the main source of heat in the thermal runaway process of the cell is the reaction between the cathode and electrolyte, and between the cathode and anode. The specific reaction enthalpies are shown in Table 3.

#### 3.2. DSC testing of battery materials with amine additives

Primary and secondary amines can induce ring-opening polymerization of five-membered cyclic

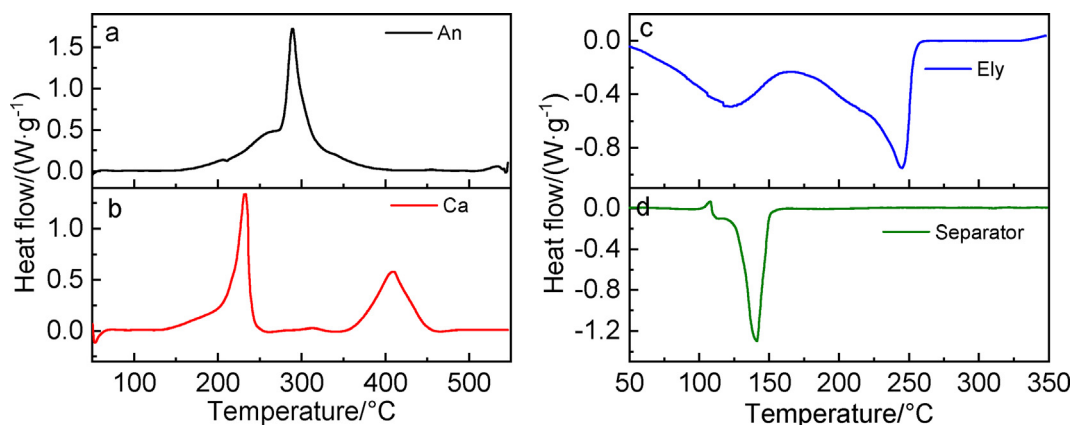


Fig. 1. DSC curves for the individual component samples: (a) Anode; (b) Cathode; (c) Electrolyte; (d) Separator.

organic compounds, Ethylene carbonate (EC), in commercial electrolytes, due to the chemical nature of their own functional groups, such as DBA, as shown in Fig. 3. The polyurethane colloidal substance, that is formed by ring-opening polymerization, can block the gas crosstalk after the cell has produced gas, and regulate the reactions sequence between the battery materials, thereby reducing the exothermic intensity of the reactions [15].

According to the number of hydrogen atoms substituted in the amine molecule, the amine additives were selected as primary, secondary and tertiary amines, i.e., 1,2-ethylenediamine (1,2-EDA), DBA and trihexylamine (THA), respectively, as the reagents to be tested. The results of DSC testing of the all components sample with amine additives are shown in Fig. 4. It is found that the addition of 4% mass ratio of 1,2-EDA, DBA and THA resulted in a significant decrease in the peak heat flow of the all components sample, by 53.8%, 67.3% and 55.8%, respectively. Specifically, the reaction onset temperature of the all components sample of the control samples was 150 °C. After the

Table 3. DSC test results for some components of the battery.

Sample composition	Onset Point (°C)	End Point (°C)	Enthalpy ( $\text{J}\cdot\text{g}^{-1}$ )	Enthalpy (50–550 °C) ( $\text{J}\cdot\text{g}^{-1}$ )
An/Ely	141	271	321.43	228.46
Ca/Ely	215	277	764.43	682.11
Ca/An	219	276	557.22	557.22
Ca/An/Ely	150	289	713.08	967.59

addition of 1,2-EDA, a significant exothermic reaction was detected at the beginning of the test, with a significant decrease in the heat flow intensity of the main exothermic peak in the 200–250 °C range. The sample with the addition of DBA showed similar exothermic properties, with an earlier reaction onset temperature of 114 °C and a decrease in the intensity of the main exothermic peak. In contrast, THA had a more stable performance, which also means that it had little effect on the TR reaction sequence.

The reaction enthalpies for DSC testing of all components and amine additives are shown in Table 4. Obviously, we can find that with the help

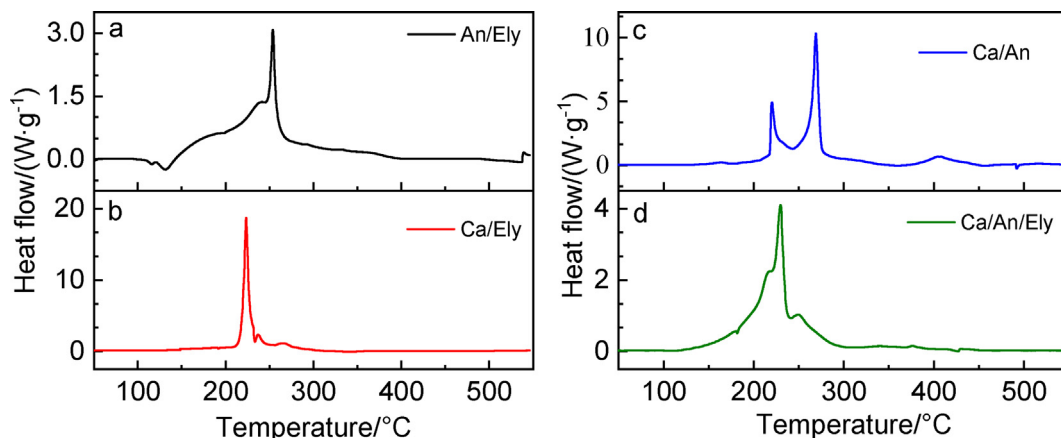


Fig. 2. DSC curves for the partial components samples: (a) Anode/Electrolyte; (b) Cathode/Electrolyte; (c) Cathode/Anode; (d) Cathode/Anode/Electrolyte.

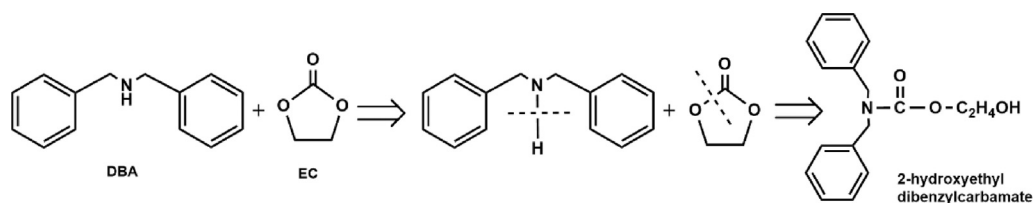


Fig. 3. Ring-opening polymerization of DBA with EC<sup>0</sup>.

of the DBA, a lower enthalpy is shown. The 1,2-EDA shows an increased enthalpy accompanied by the exothermic peak being split in two and a significant advance. And the THA also enhances the enthalpy but seems to have no effect on the starting point of the reaction.

Partial components sample testing can investigate the inhibitory effect of amine additives on key reactions during TR. This work focuses on the effect of additives on the early heat accumulation reaction (anode/electrolyte) and the TR trigger reaction (cathode/electrolyte). The experimental results are shown in Figs. 5 and 6. It is found that amine additives can effectively inhibit the cathode/electrolyte reaction and can therefore be used to inhibit the TR triggered by the violent exothermic reaction between the cathode and electrolyte (Fig. 5). In addition, the effect of the amine additive on the reaction between the anode and the electrolyte was to cause an earlier onset temperature for this reaction, because the heat is released from the ring-opening polymerization reaction of the amine with the EC (Fig. 6). Furthermore, the reaction products of the amine and EC or the amine undergo a new chemical reaction with the anode because this is not observed in the presence of an exothermic peak advance in an anode-free experiment. This may not be a bad thing. With the addition of amine additives, when the battery temperature reaches 200 °C ±, there will not be

enough anode to react violently with the cathode and thus inhibit the effects of TR.

### 3.3. ARC testing of batteries with amine additives

Based on the above experimental results, we believe that 1,2-EDA and DBA can better change the heat production characteristics and reaction sequence between battery materials. Therefore, we chose 1,2-EDA and DBA for the verification of their effects on the TR characteristics of the complete battery. The experimental SiC||NCM811 battery, that had been implanted with the thermocouple, was first fully charged. Next, the aluminum plastic film was cut open and filled with amine additives (1,2-EDA, DBA) at 4% of the battery cell mass using a pipette, and then the aluminum plastic film was sealed using a heat sealer. Finally, an ARC test was run to verify the change in TR characteristics of the SiC||NCM811 battery with amine additives under thermal abuse and the suppression effect of amine additives.

As shown in Fig. 7, the addition of the amine additives resulted in a significant reduction in the time from  $T_1$  to  $T_2$ , while the  $T_3$  of the battery showed a definite decreasing trend compared to the reference battery. Here, we define the battery without amine additives as the reference battery. In particular, 1,2-EDA reduced the maximum thermal runaway temperature  $T_3$  from 1024 °C to 668 °C, suppressing it by approximately 32.7%. DBA reduced the  $T_3$  from 1024 °C to 831 °C, suppressing it by approximately 18.7%. This result indicates that the effect of the tested amine additives on the TR behavior of the battery lay mainly in the inhibition of the maximum thermal runaway temperature. This is the result of the early

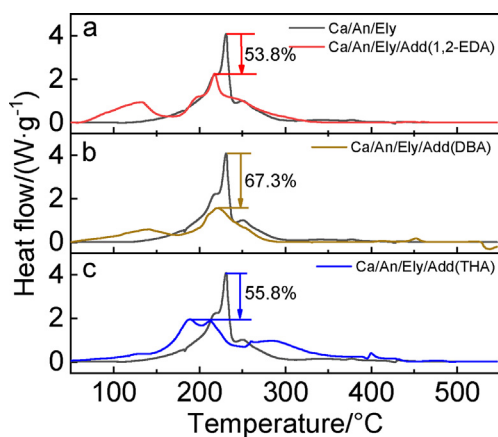


Fig. 4. DSC curves for all components sample with amine additives: (a) 1,2-EDA; (b) DBA; (c) THA.

Table 4. DSC test results for all component samples with amine additives.

Sample composition	Onset point (°C)	End point (°C)	Enthalpy (J·g <sup>-1</sup> )	Enthalpy (50–550 °C) (J·g <sup>-1</sup> )
Ca/An/Ely	150	289	713.08	967.59
Ca/An/Ely/1,2-EDA	50	295	854.19	1195.87
Ca/An/Ely/DBA	114	277	396.16	625.32
Ca/An/Ely/THA	157	340	632.13	1131.99

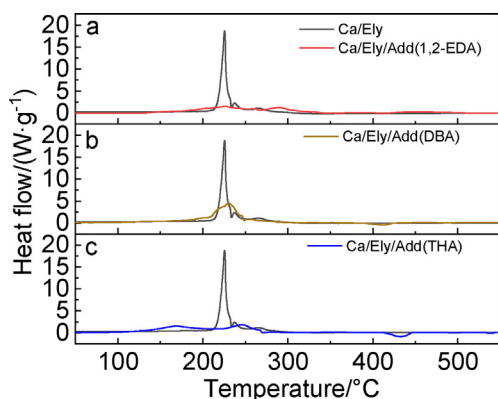


Fig. 5. DSC curves of Ca/Ely/Add: (a) 1,2-EDA; (b) DBA; (c) THA.

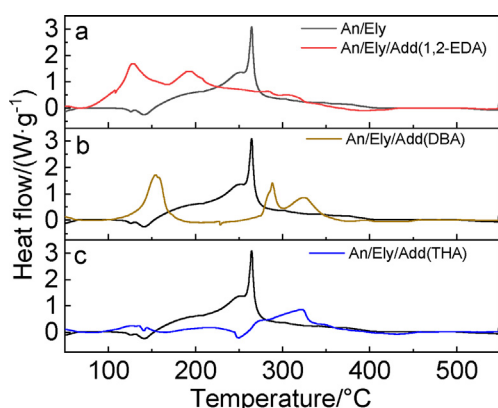


Fig. 6. DSC curves of An/Ely/Add: (a) 1,2-EDA; (b) DBA; (c) THA.

Table 5. ARC test data for batteries with amine additives.

Battery information	Amine additive	$T_1$ (°C)	$T_2$ (°C)	$T_3$ (°C)
SiC  NCM811	—	102	209	1024
5 Ah	1,2-EDA	70	220	688
	DBA	56	202	831

consumption of the anode. The TR characteristic parameters are shown in Table 5.

The temperature rise rate variation during the TR of the battery can be obtained by differentiating the ARC data, as shown in Fig. 7. The internal maximum temperature rise rate for the reference battery was  $1.26 \times 10^4$  °C/min. The maximum temperature rise rate for the battery with 1,2-EDA was  $5.02 \times 10^3$  °C/min, which is a decrease of approximately 60.2%. Furthermore, the addition of DBA to the complete battery resulted in a maximum temperature rise rate of  $1.13 \times 10^4$  °C/min, a decline of about 10.3%.

#### 4. Conclusions

In conclusion, this work provides a considering way to reduce the risk of TR in lithium-ion batteries. Amine additives had a certain inhibitory effect on the TR of the battery, which is mainly reflected in the reduction of the maximum TR temperature. The inhibition mechanism of the amine additive on the thermal properties of the battery material is

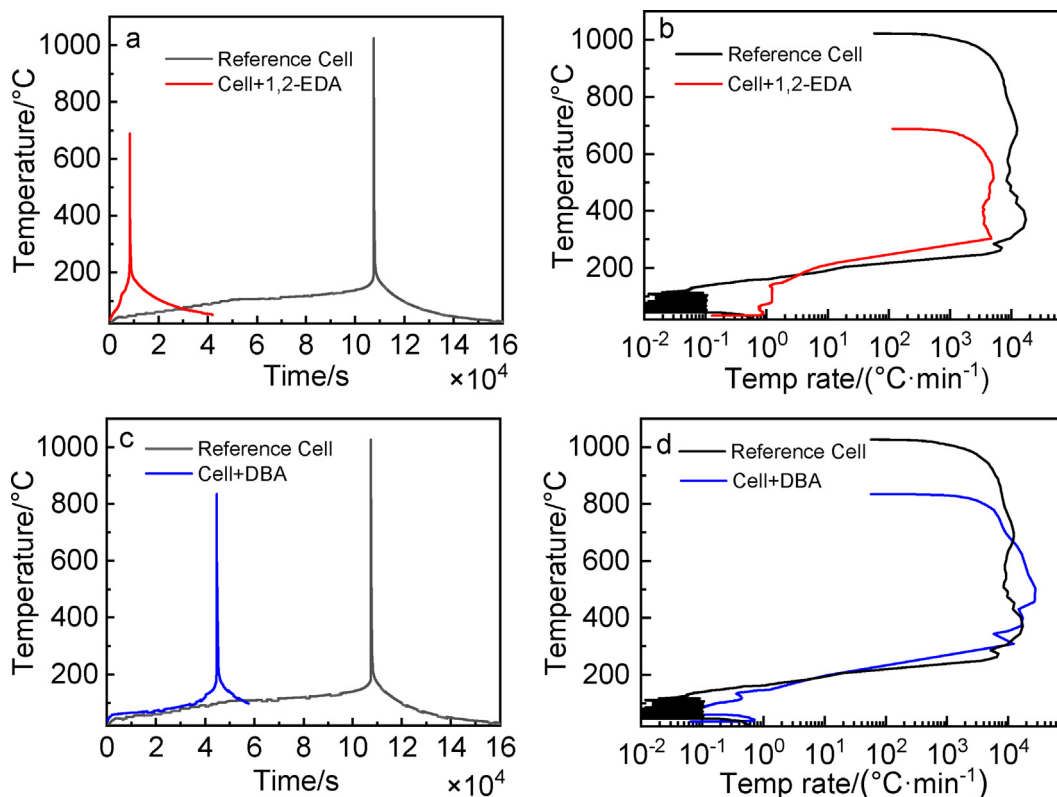


Fig. 7. ARC test and temperature rise rate variation of the complete battery with amine additives: (a) 1,2-EDA\_ARC; (b) 1,2-EDA\_temperature rise rate; (c) DBA\_ARC; (d) DBA\_temperature rise rate.

due to the induction of ring-opening polymerization of the EC, forming a polyurethane barrier to gas crosstalk within the battery. For the influence of the thermal stability of the battery material, there was an earlier exothermic reaction onset temperature, after adding a certain amount of amine additives. However, the main exothermic peak heat flow intensity decreased, while the exothermic heat was reduced by more than 50% for all components sample. The reaction products of amines and EC or amines could react with the charged anode material. When the cell temperature reached  $200\text{ }^{\circ}\text{C} \pm$ , there would not be enough anode to be able to react with the cathode so that the effects of TR could be inhibited. This was demonstrated in experiments at ARC. The inhibition of TR behavior for the complete battery lies in the reduction of the maximum TR temperature  $T_3$  and the maximum temperature rise rate  $dT/dt_{\max}$  in the case of thermal abuse, but shortens the time from the self-generated thermal temperature  $T_1$  to the TR trigger temperature  $T_2$  of the battery.

This means that the addition of amine additives to a normal battery can lead to earlier thermal runaway of the battery. However, if there is a suitable means of injecting amine additives into the battery after the self-generated thermal has already occurred, this will effectively reduce the effects of TR. Therefore, an effective coating method is particularly important.

## Acknowledgements

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## 胺类添加剂对 NCM811 || SiC 电池热失控抑制效果研究

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### 摘要

高镍三元电池的高能量密度是取代化石能源, 推动清洁能源发展的核心优势, 同时也是导致电池严重安全隐患的根本原因。初级胺类与次级胺类能与常见的含碳酸乙烯酯电解液发生开环聚合, 从而形成正负极间隔离层, 提高电池热安全性。本文基于胺类和电池组分间的化学反应, 在电池材料层面和单体层面对电池的安全性展开了研究。在材料层面, 利用差示扫描量热法测试锂离子电池中有无胺类添加剂对不同组分间的热稳定性影响。在单体层面, 使用绝热加速量热仪对有无添加剂全电池的安全性进行测试, 提取热失控特征温度。加入胺类添加剂后电池组分间部分化学反应被提前, 同时总放热量明显减少, 最大温升速率下降, 电池热失控得到有效抑制。

**关键字:** 锂电池; 热失控抑制; 胺; 电解液; 开环聚合