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Summary

Li-ion batteries have become an important part of our daily life in applications as different as cell phones and electric ferries. High energy density is one of the factors for the successful market penetration of this technology. However, when stressed beyond their design limits, Li-ion batteries can start self-heating and reach thermal runaway, releasing combustible and unhealthy gases, catching fire or even explode.

In so-called propagation tests, battery modules or installations are tested for their ability to prevent propagation of thermal runaway between cells or modules. Thermal runaway is initiated in one cell using a chosen initiation method. An important topic for propagation tests is: How should thermal runaway be initiated so that it best resembles a realistic field failure?

The Norwegian Defence Research Establishment (FFI) has investigated the safety of a cylindrical Li-ion cell with iron phosphate based chemistry and capacity in the 30–60 Ah range. During these studies, several initiation methods have been used. Results from the various experiments highlight some of the differences between initiation methods. This report summarizes the differences and provides a background for choosing a suitable initiation method.

Single cells were forced into thermal runaway using various methods of external heating (nozzle heaters, flexible heating sheet, infrared radiation heating, adiabatic heating) or by generating internal short circuits (internal heating element, nail penetration). The cell behaviour was observed and categorized according to hazard severity levels.

The cells revealed a large variation in cell behaviour, both for different initiation methods and for identical methods. All hazard severity levels between 4 (major leakage or vent) and 7 (energetic failure) were observed, and mass losses ranged from 15 to 86%. This variation shows that abuse tests or propagation tests without repetitions can give a misleading impression of the potential hazards of the battery. It also shows that a single successful safety test example is not sufficient evidence for considering a cell as safe. Repeated testing is necessary to reveal all possible cell behaviours.

Out of the tested methods for initiating thermal runaway, internal heating element was the method that gave least variation in cell behaviour. This method generally did not produce the worst-case cell behaviour observed for many of the external heating techniques.

Module developers and safety evaluators should be aware of the possibility for sidewall rupture when using cells with rigid walls. Ruptures can cause the ventilation gases to be released in unintended directions. The results also clearly demonstrated the flammability of the released gases and the possibility for ignition. Additionally, the results exemplified that cell wall temperature measurements cannot be regarded as a reliable pre-warning parameter for thermal incidents.

Sammendrag

Li-ionbatterier har blitt en viktig del av hverdagslivet i anvendelser så ulike som smarttelefoner og elektriske ferger. Høy energitetthet er en av faktorene for at teknologien har lykkes i så mange markeder. Når Li-ionbatterier utsettes for større påkjenninger enn de er designet for, kan de imidlertid begynne å selvoppvarme og havne i en selvforsterkende overopphetingsreaksjon kalt thermal runaway. Ved thermal runaway slipper batteriet ut brennbare og helseskadelige gasser, det kan ta fyr eller til og med eksplodere.

I såkalte propageringstester blir batterimoduler eller -installasjoner testet for sin evne til å hindre propagering av thermal runaway mellom celler eller moduler. Thermal runaway blir igangsatt i en celle med en valgt initieringsmetode. Et viktig emne for propageringstester er: Hvordan bør thermal runaway initieres slik at det ligner mest mulig på en realistisk feilsituasjon?

Forsvarets forskningsinstitutt (FFI) har undersøkt sikkerheten til en sylindrisk Li-ioncelle med jernfosfatbasert kjemi og kapasitet i området 30–60 Ah. I disse studiene er en rekke ulike initieringsmetoder blitt brukt. Resultater fra de ulike eksperimentene understreker noen av forskjellene ved initieringsmetodene. Denne rapporten oppsummerer forskjellene og gir et informasjonsgrunnlag for å velge passende initieringsmetoder.

Thermal runaway ble framprovosert i enkeltceller ved hjelp av ulike former for ekstern oppvarming (dysevarmere, fleksible varmeark, infrarød strålingsovn, adiabatisk oppvarming) eller ved å generere interne kortslutninger (internt varmeelement, spikerpenetrering). Celleoppførselen ble observert og kategorisert i henhold til en skala for farenivå.

Cellene viste en stor variasjon i oppførsel, både for ulike initieringsmetoder og for identiske metoder. Alle farenivå mellom 4 (stor lekkasje eller ventilering) og 7 (energetisk feil) ble observert, og massetap varierte fra 15 til 86 %. Denne variasjonen viser at misbrukstester eller propageringstester som ikke blir repetert, kan gi et feilaktig inntrykk av de mulige farene ved batteriet. Den viser også at et enkeltstående eksempel på en vellykket sikkerhetstest ikke er tilstrekkelig grunnlag for å betrakte en celle som sikker. Gjentatt testing er nødvendig for å avdekke alle mulige celleoppførsler.

Av de testede initieringsmetodene var internt varmeelement den metoden som gav minst variasjon i celleoppførsel. Denne metoden forårsaket i all hovedsak ikke den kraftigste cellereaksjonen som ble observert for mange av teknikkene med ekstern oppvarming.

Modulutviklere og sikkerhetsinspektører bør være klar over muligheten for sideveggsrevning når celler med stive vegger blir brukt. Revner kan føre til at ventilerte gasser slippes ut i utilsiktede retninger. Resultatene demonstrerer også tydelig at gassutslippet er brennbart og muligheten for antenning. I tillegg gir resultatene eksempler på at målinger av celleveggtemperatur ikke kan anses som en pålitelig parameter for varsling av termiske hendelser.

Contents

Preface

The authors would like to acknowledge the Research Council of Norway for providing funding for writing this report, through the Norwegian Research Centre Mobility Zero Emission Energy Systems (MoZEES). We would also like to acknowledge RISE Fire Research in Trondheim for assisting in some of the abuse testing reported here. Our colleague Sissel Forseth is acknowledged for valuable comments to the report.

Kjeller, 28 June 2021
Helge Weydahl, Kjetil Valset, Martin Gilljam, Torleif Lian, Knut Bjarne Gandrud

1 Introduction

Li-ion batteries have become an important part of our daily life in applications as diverse as cell phones and electric ferries [1, 2]. High energy density is one of the factors for the successful market penetration of this technology. However, high energy density also introduces a safety issue: When stressed beyond their design limits, Li-ion batteries can start self-heating, release flammable and unhealthy gases, catch fire or even explode [3, 4]. The process where internal self-heating accelerates irrevocably and the temperature increase rate exceeds 10 ºC/min, is called thermal runaway.

Most causes of thermal runaway in Li-ion batteries can be prevented with proper battery management systems and battery design. However, some incidents can stem from internal short-circuits, which can cause spontaneous cell fires despite a well-designed battery management system [5]. In so-called propagation tests, battery modules or installations are tested for their ability to restrain such incidents. Using a thermal runaway initiation method, one cell is forced into thermal runaway, and the propagation to neighbouring cells or modules is investigated. In propagation tests, the initiation method should replicate a cell behaviour corresponding to an internal short circuit, since such failures cannot be averted by the battery management system.

There are several methods available to force a cell into thermal runaway. Abuse tests are designed to characterize the battery behaviour during overheating, overcharge, short circuit, mechanical impact etc. [6-8]. Available abuse tests and thermal runaway initiation methods have been summarised in recent papers [6, 7, 9-11]. During an abuse test, the cell is stressed beyond its design limits, so violent reactions can be expected. All of the available methods have advantages and disadvantages in mimicking a real cell failure. An important and recurring topic for propagation tests is: How should thermal runaway be initiated so that it best resembles a realistic field failure?

The Norwegian Defence Research Establishment (FFI) has investigated the safety of a cylindrical Li-ion cell with capacity in the 30–60 Ah range. During these studies, several thermal runaway initiation methods have been used. Results from the various experiments highlight some of the differences between initiation methods. This report summarizes the differences and provides a background for choosing a suitable initiation method.

It is important to emphasize that the original intention of the experiments was not to compare initiation methods. The experiments were not designed equivalently and therefore do not give a scientifically sound basis for comparison. However, the results still give insight into the diverse cell behaviour resulting from abuse testing with different initiation methods. For this reason, the report focuses on the visual outcome of the tests in terms of damage to the cell, characteristics of gas release, ejection of cell material etc.

This report is organised as follows: We first describe the cell used for the experiments, and the hazard severity levels used to categorize their behaviour during abuse tests. Then, we go through the various initiation methods, with experimental set-up and results. Finally, we discuss the main findings from the experiments and then conclude.

2 Cell description

The experiments in this report have been carried out using a cylindrical cell with capacity in the 30–60 Ah range. The cathode chemistry is iron phosphate based. The sidewall of the cell is covered with plastic shrink-wrap, but this was removed before most of the experiments. The cell has a device, which opens at a certain internal pressure. Further details about the cell cannot be revealed due to a non-disclosure agreement with the manufacturer. For the same reason, no pictures or illustrations of the cell is included in this report.

FFI has tested three versions of this cell, designated version 1, 2 and 3. These versions have small differences in cell contents and up to 7% difference in capacity. Therefore, the experiments referred to in this report are not strictly comparable according to scientific standards. This must be kept in mind when evaluating differences between the results.

Results in this report were all obtained with cells at 100% state of charge (SoC). Some of the cells were cycled four times before they were charged. Further details of the cell treatment before experiments are given in the sections below.

3 Hazard severity levels

In this report, we rate the cell behaviour during abuse tests using hazard severity levels (HSLs). These were defined by Sandia National Laboratories in 2017 [8] and are shown in Table 3.1. It is important to note that this scale is different from the EUCAR Hazard level which was defined by Sandia National Laboratories in 2005 [12]. A comparison of the two scales is shown in Appendix A. We have used the HSLs of 2017 in this report since they represent the most updated version. They are also provided with more quantifiable parameters, which aids in a more precise categorization of the results.

Table 3.1 HSL rating table reproduced from Sandia report 2017-6925 [8].

Hazard Severity Level	Description	Classification Criteria and Effect
0	No effect	No effect. No loss of functionality.
1	Passive protection activated	No damage or hazard; reversible loss of function. Replacement or re-setting of protection device is sufficient to restore normal functionality.
2	Defect/Damage	No hazard but damage to the rechargeable energy storage system (RESS); irreversible loss of function.
3	Minor Leakage or Minor Vent	Visual or audible evidence of leaking or venting. Leak without significant pooling or collection of free liquid. Venting without significant smoke or loss of particulate material. No visual obstruction of the RESS.
4	Major Leakage or Major Vent	Visual evidence of leaking or venting. Leaking with significant pooling or observed free liquid. Venting with significant smoke, solvent vapor, and/or loss of particulate material. Visual obstruction of the RESS by vent gases and/or smoke. Total RESS mass loss < 30%.
5	Rupture	Loss of mechanical integrity of the RESS package, resulting in release of contents. The kinetic energy of released material is not sufficient to cause physical damage external to the RESS. Rupture *may* be the result of a RESS thermal runaway (but not necessarily). Total RESS mass loss 30–55%
6	Fire or Flame	Ignition and sustained combustion of flammable gas or liquid (≥1 s sustained fire). Sparks or incandescent material is not considered a fire or a flame.
7	Energetic Failure	Fast release of energy sufficient to cause pressure waves (slower than the speed of sound) and/or projectiles that may cause considerable structural and/or bodily damage, depending on the size of the RESS. The kinetic energy of flying debris from the RESS may be sufficient to cause damage as well. Total RESS mass loss ≥ 55%.

Table 3.1 does not specify how results should be categorized if only some classification criteria at a certain level are fulfilled. In this report, we have used an approach, where results are rated

at the highest HSL where at least one classification criterion is fulfilled. As an example, a cell that ruptures is considered as HSL 5 even though the mass loss is less than 30%.

HSL 7 requires an energetic failure with mass loss above 55%. Since we had limited means to evaluate the kinetic energy of projectiles or flying debris, it was not possible to use this criterion to distinguish between level 5 and 7. This means that only cells with mass loss above 55% are categorized as HSL 7 in this report.

HSL is a useful tool to categorise cell behaviour during abuse tests. However, when evaluating the safety of a specific battery module, the way in which the abuse response of the cell is managed by the module's safety design is more important than the cell's HSL. As an example, HSL 6 (fire or flame) may not be a more severe safety hazard than HSL 4 (major vent) if the module is designed for handling fire in a safe manner.

4 Initiation methods

4.1 Nozzle heaters 2 ºC/min

4.1.1 Experimental set-up

The cells were initially cycled four times at C/5 and charged to 100% SoC according to the manufacturer's specifications. The cell was inserted into an aluminium pipe with inner diameter closely matching the outer diameter of the cell, see Figure 4.1. Before inserting the cell, the plastic shrink-wrap was removed to ensure thermal contact with the pipe. The pipe was externally heated by three 380 W nozzle heaters. The temperature in the middle of the aluminium pipe wall was measured, and the heater current was regulated to obtain a constant heating rate of 2 ºC/min. The pipe wall was insulated, but the top and bottom end was uninsulated.

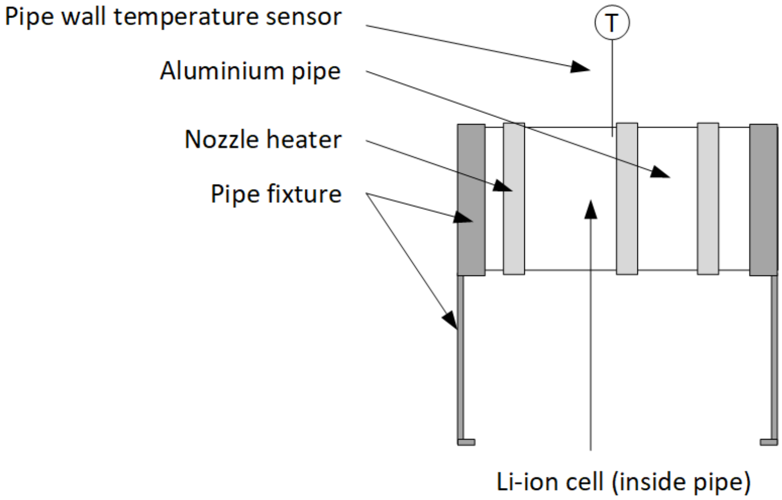

Figure 4.1 Side view of experimental set-up for experiments with external heating at 2 ºC/min. Pipe insulation is not shown. The drawing is not to scale.

The intention of the set-up and the chosen heating rate was to obtain slow homogeneous heating of the cell until thermal runaway occurred. The aluminium pipe distributes the heat across the cell wall, but some temperature gradients can be expected near the cell end lids.

One heating experiment was carried out outdoors and four in a sealed container with nitrogen atmosphere. Versions 1 and 2 of the cell were tested in these experiments. In the outdoor experiment, the initial cell temperature was 23 ºC, and the ambient temperature was 16 ºC. In the sealed container, the initial temperature was 30 ºC, equal to the wall temperature of the sealed container.

4.1.2 Results

The cell behaviour during the experiments showed the following features: First, the cell voltage fell to 0 V at a pipe wall temperature range of 143–146 ºC. Then the pressure relief device opened with an audible noise in the temperature range 156–161 ºC, releasing gases from the cell. The gas release rate increased gradually towards the point of thermal runaway, which occurred when the pipe wall temperature was in the range 196–204 ºC. This is considerably lower than the thermal runaway temperature measured on the cell wall during accelerating rate calorimetry experiments (ref. Chapter 4.7). We explain this difference by the thermal inertia of the aluminium pipe. During thermal runaway, the cell released gases and ejected varying amounts of its material in one or several audible bursts. Video recordings of the outdoor experiment showed that also sparks were ejected from the cell during thermal runaway, and a brief ignition (<0.1 s duration) of the gas cloud was observed. Gas release subsided approx. 1 min after thermal runaway and smoke was no longer visible after 5 min.

Inspection after the tests revealed no ruptures in the sidewall of the cell. The amount of cell material that was ejected varied from 23 to 62% of the cell's original mass. Considering the mass loss limits in Table 3.1, the cells exhibited HSLs 4, 5 and 7. Even though sparks and short-lived ignition were observed in the outdoor experiment, this does not qualify for HSL 6 (fire or flame). In the sealed container, we did not have the possibility to inspect for fire or flame visually. In addition, the container was filled with nitrogen, further reducing the chance for fire or flame.

A low heating rate of 2 ºC/min is not typical for a thermal event where the cell eventually reaches thermal runaway. It can however correspond to a scenario where the cell is heated slowly by a small internal short circuit, by failing neighbouring cells, by overload or by an external fire. We note that at this heating rate, the cell voltage dropped 8 min before the pressure relief device opened. As for most cylindrical and prismatic cells, the investigated cell most likely contains a current interrupt device that breaks the electrical connection to the terminals when the internal pressure exceeds certain levels [13]. This will affect the measured cell voltage, but we do not know if the observed cell voltage drop was caused by such a device.

The advantage with this method is that we obtain more homogeneous temperature conditions and less variation between tests than for a high heating rate: Cell voltage drop, ventilation and thermal runaway occurred at nearly the same temperatures in all tests. The main weakness is that it does not represent the typical behaviour for a cell approaching thermal runaway, where higher heating rates can be expected.

It is important to note that in this method, as well as the other methods using external heating (see Chapters 4.2, 4.3 and 4.4), the cell is forced to higher temperatures even though the pressure relief device opens. In a real thermal event, ventilation could cause the internal self-heating to stop so that the cell does not reach thermal runaway. We did not investigate if the cell was able to reach thermal runaway if the external heaters were turned off at ventilation.

4.2 Nozzle heaters 15 ºC/min

4.2.1 Experimental set-up

The experimental set-up was identical to the one described in Chapter 4.1.1, but to obtain a higher heating rate, six nozzle heaters were used instead of three, as shown in Figure 4.2. With this set-up, the aluminium pipe wall temperature increased with 15 ºC/min.

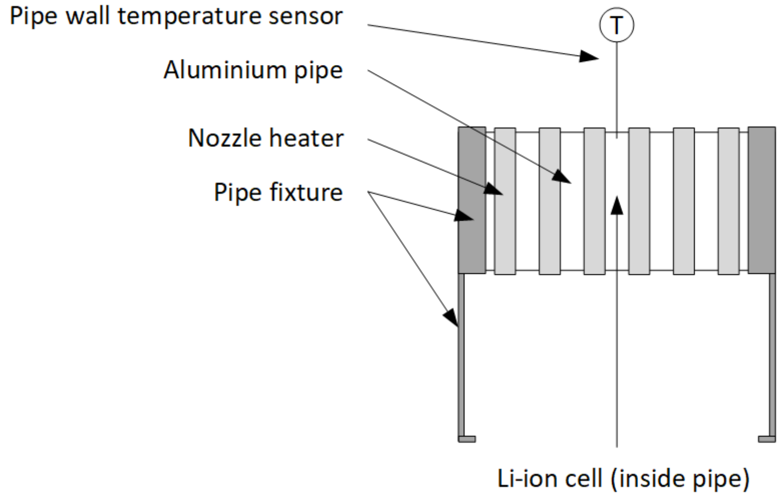

Figure 4.2 Side view of experimental set-up for experiments with external heating at 15 ºC/min. Pipe insulation is not shown. The drawing is not to scale.

The intention of this set-up was to obtain a heating rate that is more representative of a realistic thermal event where the cell eventually reaches thermal runaway. Another intention was to observe if a higher heating rate gave different cell behaviour. With this heating rate, we expect larger temperature gradients in the cell both from the cell wall to the cell interior and from the centre towards the ends. We do not expect a homogeneous temperature distribution using this method.

Two outdoor experiments were carried out at this heating rate, using cells of version 2 and 3. For these experiments, the initial cell temperature was 24 and 20 ºC, respectively, and the ambient temperature was 12 and 7 ºC. In addition, seven experiments were carried out in nitrogen atmosphere inside a sealed container, three with version 2 cells and four with version 3 cells. The initial cell temperature was 30 ºC for these experiments, equal to the wall temperature of the sealed container.

4.2.2 Results

The cell behaviour during the experiments showed the following features: The pressure relief device opened with an audible noise in the pipe wall temperature range 218–236 ºC. Gases were released at a higher rate than for the experiments with 2 ºC/min heating rate. The cell voltage dropped 0–50 s after ventilation. The gas release rate increased gradually towards the point of thermal runaway, which started at pipe wall temperatures in the range 266–306 ºC. During thermal runaway, the cell released gases and ejected varying amounts of its material in one or several audible bursts. In the outdoor experiments, sparks and burning cell material ejected from the cell were seen to ignite the released gases. In one instance, flames from burning cell material was visible for 17 s. The kinetic energy of flying debris was varying: In some cases, only a sharp hissing noise could be heard from the sealed container during thermal runaway. In other cases, the cell reaction was sufficiently forceful that the aluminium pipe made a somersault or came loose from the fixture. This occurred both in one outdoor experiment and in the sealed container. In the outdoor experiments, gas release subsided within one minute after thermal runaway and smoke was no longer visible after 3–8 min.

Inspection after the tests revealed no ruptures in the sidewall of the cell. The amount of cell material that was ejected varied from 21 to 83 % of the cell's original mass. On one occasion, the cell opened in both ends.

Considering the mass loss, the cells exhibited HSLs 4, 5 and 7. Note that the cell ejecting burning cell material was categorized as HSL 7 instead of 6 due to its high mass loss. As in Chapter 4.1, we did not have the possibility to inspect for fire or flame visually in the nitrogen-filled container.

Compared to 2 ºC/min, a heating rate of 15 ºC/min is closer to what can be expected for a cell approaching thermal runaway. In addition, the temperature gradients in the cell will be larger using this method. It should be noted that ventilation always occurred before or at the same time as the cell voltage dropped when a heating rate of 15 ºC/min was used. As mentioned, a current interrupt device will affect the measured cell voltage, and we do not know if such a device or other effects caused the observed drop.

4.3 Flexible heating sheet

4.3.1 Experimental set-up

The cell was initially cycled four times at C/5 and charged to 100% SoC according to the manufacturer's specifications. In this set-up, a flexible heating sheet from OMEGA™ was fastened directly to the cylinder sidewall of the cell using glass fibre tape, after first removing the plastic shrink-wrap around the cell. The heating sheet was wrapped around the cell, covering 61% of the sidewall area. The cell was put on top of an insulation sheet in one of the shelves in a temperature chamber at 30 ºC. The cell was otherwise uninsulated. The cell wall temperature was measured with a thermocouple positioned at the centre of the cell's sidewall in an area section that was not covered by the heating sheet, facing the insulation sheet. The thermocouple was fastened to the cell wall with glass fibre tape. The cell was heated with maximum available power (140 W) which was sufficient to obtain a heating rate of around 5 ºC/min measured on the sidewall of the cell.

One of the intentions with this experiment was to investigate external heating with a slimmer heating device than nozzle heaters. Only one experiment was carried out, using a cell of version 2.

4.3.2 Results

The cell behaviour during the experiment showed the following features: The cell voltage started to decrease and made a sharp drop to 0.4 V at a cell wall temperature of 153 ºC. The pressure relief device opened with an audible noise at a cell wall temperature of 168 ºC. Thermal runaway started at 256 ºC. Since the experiment took place inside a temperature chamber, the cell behaviour during thermal runaway could not be visually observed. However, white smoke came out of the chamber and two bursts could be heard. Gas release subsided within a minute after thermal runaway.

Cell inspection after the experiment revealed that the sidewall had ruptured and cell material had been pushed out through the rupture. The cell material formed a plume protruding 1-2 cell diameters out from the rupture. The cell lost 26% of its original mass during the experiment. This is less than the mass loss limit for HSL 5. We still categorize this as HSL 5 due to the undisputable loss of mechanical integrity.

The advantage of this method is the slim shape of the heater. This makes it more suitable for propagation tests in battery modules where the available space around the cells is limited. The described experiment also clearly illustrated the disadvantage of this method: A heating sheet directly attached to the cell wall can affect the outcome of the test by weakening the cell wall. In this experiment, the heating sheet was most likely a contributing factor to the sidewall rupture.

4.4 Infrared radiation heating

4.4.1 Experimental set-up

These experiments were performed at the facilities of RISE Fire Research in Trondheim. The cells were charged with a constant current, constant voltage regime (C/8.8 to the upper voltage limit, constant voltage until the charging current dropped below C/23). The plastic shrink-wrap around the cell was removed.

The intention of these experiments was to measure the energy released during thermal runaway using cone calorimetry. The cell was positioned horizontally within a steel container with bottom and sidewalls consisting of 1.5 mm steel plates, and a top wall consisting of a steel mesh (with 10 mm x 20 mm openings). The cell was positioned within the steel container using steel pipe clamps at each end of the cell, and elevated from the bottom of the steel container with a steel rod. The steel rod and clamps were electrically and thermally insulated from the cell by 2 mm non-combustible insulation. The set-up is illustrated in Figure 4.3.

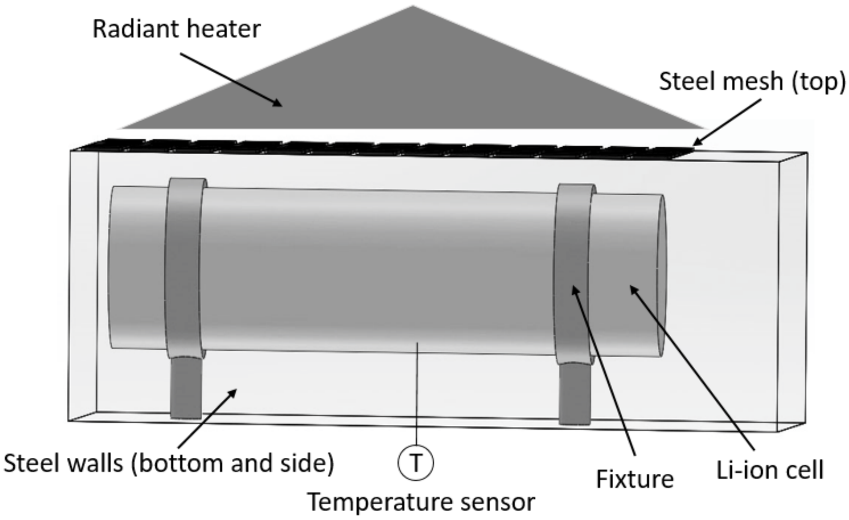

Figure 4.3 Experimental set-up for experiments with infrared radiation heating. The drawing is not to scale.

The cell was heated by an electrical cone-shaped radiant heater positioned 30 mm above the cell. The radiant heat flux was 41 kW/m^2 resulting in topside heating with rates between 4 and 6 ºC/min, until thermal runaway occurred. The heater also acted as an ignition source for the gases released from the cell. The cell temperature was measured with a copper disc thermocouple attached to the middle of the cell sidewall facing away from the heater. The experiments were performed at ambient temperatures around 18 ºC.

Three experiments with this set-up were carried out using cells of version 3.

4.4.2 Results

The cell behaviour during the experiments showed the following features: First, the cell voltage started to decrease, either sharply or gradually, when the mid cell wall temperature reached 147–150 ºC. A video recording of one of the experiments showed that the pressure relief device opened at 151 ºC, releasing gases from the cell. The gases were ignited by the heater 4 min after ventilation and burned for another 5 min before thermal runaway occurred. In the other two experiments, thermal runaway was reached 4–9 min after the first flames were observed. The mid cell wall temperature at the beginning of thermal runaway was in the range 221–236 ºC. During thermal runaway, a loud noise and a fireball were observed. Sparks or embers were flying. Flaming continued for 3–5 min after thermal runaway.

Inspection after the tests revealed that the cells had ruptured and disintegrated. Only the part of the cell wall that was supported by the clamps was still intact. The amount of cell material that was ejected was not measured for these tests. Due to the presence of continuous flames, all cells were categorized as HSL 6.

The described method produced three very similar outcomes with flames and disintegration of the cells. The radiant heater acted as a permanent ignition source for the ejected gases. This caused flaming for several minutes. Flames were also observed in one case when using nozzle heaters (see Chapter 4.2.2), but this lasted for only 17 s.

Both the radiant heater and the flames around the cell before thermal runaway can weaken the cell wall. As for the flexible heating sheet in Chapter 4.3, this can affect the outcome of the test. Consequently, radiant heating appears to be less suitable as a method to characterize the cell behaviour during a thermal event.

4.5 Internal heating element

4.5.1 Experimental set-up

Cells of version 3 were used for these experiments. A custom-made internal heating element was installed inside the cell by the cell manufacturer. The heating element was supplied by electric wires through a connection on the top lid of the cell. When the heater current was turned on, the heating element heated a small section of the jellyroll, creating an internal short circuit in the cell. The total energy supplied to the heating element amounted to less than 0.2% of the total electric energy of the cell. After the internal short circuit was generated, the heater current was turned off and the cell continued to self-heat until ventilation and thermal runaway. Further details about the internal heating element cannot be provided due to a non-disclosure agreement with the manufacturer.

Due to safety restrictions, the cells were not cycled before the experiment, but charged once to 100% SoC according to the manufacturer's specifications.

Cells with internal heating element were tested in several set-ups:

- Five cells were tested outdoors with the plastic shrink-wrap on as received. The cells were otherwise uninsulated. Ambient temperatures were between 14 and 18 ºC. The cell wall temperature was measured with a thermocouple fastened with glass fibre tape to the centre of the cell's sidewall.

- One cell was tested outdoors inside the same aluminium pipe as described in Chapter 4.1. As for the experiments in Chapter 4.1, the plastic shrink-wrap was removed before the cell was inserted into the pipe. The aluminium pipe was not insulated, but heated to a stable 30 ºC before the experiment started. The ambient temperature was 13 ºC during this test.

- Four cells were tested in a nitrogen environment at 30 ºC in a sealed container. The cells were placed inside the same aluminium pipe as described in Chapter 4.1 with their plastic shrink-wraps removed. The pipe was uninsulated and the nozzle heaters were not used. The internal heating element failed in one of these experiments and did not initiate a thermal event.

- Three cells were tested at the facilities of RISE Fire Research in the same set-up as described in Chapter 4.4, but without using the infrared radiation heater. Instead, a pilot flame for ignition of released gases was used. The plastic shrink-wrap around the cells was removed before the experiments, in line with the experiments in Chapter 4.4. In one of these experiments, the internal heating element failed and did not initiate a thermal event. The ambient temperature was between 9 and 13 ºC.

- Two cells were tested outdoors in an in-house built calorimeter. This is the same set-up as described in Chapter 4.6, but without using the nail penetration apparatus. The ambient temperature was around 16 ºC.

4.5.2 Results

The cell behaviour during the experiments showed the following features: The cell voltage started to decrease less than 0.5 min after the internal heating element was activated. The pressure relief device opened 0.5–1 min after activation. At this point, the mid cell wall temperature had increased by less than 10 ºC, and the temperature in the pipe wall showed no increase. In the outdoor experiments, liquid electrolyte was seen to drip or pour out when the pressure relief device opened. Gas release increased gradually. Thermal runaway, causing sidewall rupture in most cases, occurred approx. 1.5–3 min after activation. The cell wall temperature at this point was between 45 and 100 ºC. Cell behaviour during thermal runaway showed some variation between the experiments: increasing gas release, gases coming out in new directions due to sidewall rupture, sometimes a sharp noise towards the end of thermal runaway. No sparks were observed in the outdoor experiments. Gas release subsided 1–3 min after thermal runaway and was no longer visible after approx. 20 min.

The mass loss varied much less than for the other methods. Out of the 11 experiments where mass loss was measured, 10 cells had a mass loss in the range 15–18%. Very little or no cell material was ejected except gases and liquid electrolyte.

Sidewall rupture was observed in 10 out of 13 experiments. These were categorized as HSL 5 even though the mass loss was lower than 30%. The cells without rupture were categorized as HSL 4. In the two experiments with a pilot flame above the cell, released gases ignited shortly after ventilation and burned until thermal runaway occurred with larger flaming. These cells were categorized as HSL 6. It is worth noting that even though the cells were exposed to flames for several minutes, they did not disintegrate as the cells in Chapter 4.4, but ruptured along the sidewall only. This indicates that the infrared heating source was the main reason for the disintegration observed in Chapter 4.4. In addition, the sidewall rupture could have contributed to the difference in behaviour, providing an additional route for pressure relief.

The exceptions to the most typical behaviour described above were the following:

- No sidewall rupture was observed for the two experiments performed in the in-house built calorimeter. This can be explained by the tight enclosure providing extra mechanical support.

- In one of the calorimetry experiments, the cell opened in both ends and lost 52% of its mass. Material was ejected with sufficient force to lift the calorimeter briefly from the ground.

- In one of the experiments where the cells were contained in an aluminium pipe, the cell also opened in both ends, but lost only 18% of its mass. The sidewall ruptured for this cell.

Considering the exceptions above, it is evident that the sidewall rupture gives additional pressure relief, which can reduce the risk for energetic cell material ejection.

As a general trend, the method gave more consistent results than for the other methods. The mass loss was nearly the same for all but one experiment, and the HSL was 5 in 9 out of 13 experiments. The case where the calorimeter was lifted briefly from the ground indicates material ejection with high kinetic energy, but as discussed in Chapter 3, we cannot determine if this is sufficient to qualify for HSL 7.

4.6 Nail penetration

4.6.1 Experimental set-up

The cells were initially cycled four times at C/5 and charged to 100% SoC according to the manufacturer’s specifications. The cell was put in an in-house built calorimeter shown in Figure 4.4. Before inserting the cell, the plastic shrink-wrap was removed to ensure thermal contact with the calorimeter. The calorimeter consists of a pipe shaped aluminium enclosure tightly clamping the cell. Temperature sensors were positioned in the aluminium enclosure, recording the temperature increase rate during the experiment. The aluminium enclosure was insulated and positioned vertically. Thermal runaway was initiated by pressing a steel nail with 3 mm diameter radially into the cells with an actuator. The nail was inserted through holes in the aluminium enclosure near the middle of the cell or near the quarter length of the cell closer to the pressure relief device. Two different nail lengths were used: either a short nail with a penetration depth of 19% of the cell radius, or a long nail penetrating 56% of the radius. Three combinations of nail length and position were tested: Long or short nail at the middle position, or short nail at the quarter length position. The experiments were carried out outdoors with ambient temperatures between 5 and 27 ºC.

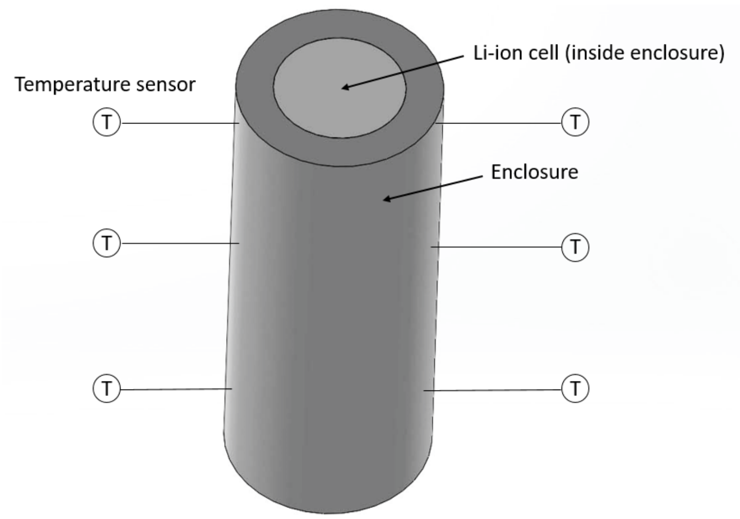

Figure 4.4 Experimental set-up for the nail penetration experiments. The cell is triggered into thermal runaway by inserting a nail into the cell through a hole (not shown) in the surrounding enclosure. The drawing is not to scale.

The intention of these experiments was to measure the heat release from the cell to the surrounding enclosure during thermal runaway. Eight cells were triggered into thermal runaway by nail penetration and the resulting heat release from the cell was measured.

4.6.2 Results

The cell behaviour during the experiments showed the following features: The pressure relief device of the cell opened 1–16 s after the nail was inserted, releasing gases from the cell. The first disturbance in cell voltage was observed 1–36 s after nail penetration. 6–37 s after nail penetration, the cell reached thermal runaway, ejecting various amounts of its cell material. The gas release after thermal runaway continued for 1–9 min. For three of the cells, the gas release started to increase again 1–2 min after thermal runaway, before it subsided. The observed differences in behaviour could not be unambiguously related to the differences in nail length and position.

Two of the cells opened in both ends (using a long nail near the middle of the cell), and the impulse from material ejection caused the calorimeter to leap from the ground. The amount of cell material that was ejected was not measured for these two cells, but for the other cells, the mass loss varied from 20 to 41%. Based on the mass loss and the state of the cells after the test, the cell behaviour using this method was categorized as HSL 4 or 5. The leaping calorimeter indicates material ejection with high kinetic energy, but as discussed in Chapter 3, we cannot determine if this is sufficient to qualify for HSL 7.

When the nail was positioned near the middle of the cell, a more rapid and larger temperature increase was observed. Apart from this general trend, the temperature evolution varied with different nail positions and lengths, and even between experiments with identical conditions. In one case (short nail positioned near the cell mid), the heat release rate was particularly low, and continued for 3.5 hours. The heat release in this case started to decrease after about 80 s. In the other experiments, the heat release started to decrease 20–55 s after nail penetration.

This method caused thermal runaway in only 6–37 s, even faster than for the experiments with internal heating element. The method caused ventilation and thermal runaway with ejection of cell material in all cases. With respect to temperature evolution, the method produced unpredictable results with heat release durations ranging from 25 min to 3.5 hours.

As other studies have pointed out [14-16], many parameters can be changed with this method. Both nail material, length, thickness, position, penetration speed etc. affect the outcome of the test. This illustrates that the nail penetration method can be manipulated by manufacturers to overrate the safety of a cell.

4.7 Accelerating rate calorimetry

4.7.1 Experimental set-up

The cells were initially cycled four times at C/5 and charged to 100% SoC according to the manufacturer's specifications. After charging, the cells were mounted vertically, with the plastic shrink-wrap on, in an aluminium cell holder, which clamped the mid-section of the cell. The cell was insulated from the cell holder using 13 mm non-combustible insulation. A thermocouple was attached to the centre of the cell's sidewall with glass fibre tape. The holder and cell were positioned inside an EV+ accelerating rate calorimeter (ARC) from Thermal Hazard Technology. The ARC was placed inside a safety chamber for protection of the laboratory surroundings.

Accelerating rate calorimetry is a method to measure the cell's self-heating rate with high precision in a semi-adiabatic environment [17, 18]. It is not regarded as an initiation method for abuse tests, in contrast to the other methods reported here.

The ARC uses a heat-wait-seek procedure, where the cell is heated in 5 °C steps from a starting temperature of 50 °C. At each step, the instrument waits for 70 min to establish equilibrium. After the wait period, the system enters seek mode where it searches for exothermic activity with a sensitivity of 0.02 °C/min. If exothermic reactions are not detected, the cycle is repeated until they are. The system then enters exothermic mode, where the self-heating of the cell is tracked under adiabatic conditions. The calorimeter follows the heating of the cell so that no heat is lost to the environment. The ARC can follow a heating rate of up to 15 °C/min. If the exothermic reaction inside the cell halts, the step-wise heating phase restarts. This continues either to the occurrence of the next exothermic response, or to the maximum achievable temperature of the calorimeter (250 °C). At temperatures above 250 °C, the ARC enters cooling mode where the chamber is flushed.

Three experiments were carried out with cells of version 2. In these experiments, the ARC chamber was initially filled with air, and the chamber was flushed with air in cooling mode. Another three experiments were carried out with version 3 cells. In these experiments, the oxygen concentration in the chamber was initially reduced by flushing with argon, and the chamber was flushed with nitrogen in cooling mode. The differences in cell versions and ambient conditions may affect the observed cell behaviour during thermal runaway.

4.7.2 Results

The cell behaviour during the experiments showed the following features: Cell voltage dropped in the cell wall temperature range 131–136 ºC (only measured for the version 3 cells). The pressure relief device opened when the cell wall temperature was 136–137 ºC. For all cells, self-heating stopped after ventilation, so that the ARC returned to the heat-wait-seek procedure until self-heating restarted in the temperature range 141–157 ºC. At thermal runaway, which started at 236–247 ºC*,* the cells released smoke and varying amounts of its cell material. Sounds from material ejection were heard from the ARC. The total mass loss varied from 28 to 67%. For the cell with highest mass loss, marks on the ARC interior indicated flying cell parts with high speed. The cylindrical cell can was pealed open in one case; in the remaining cases the sidewall was intact. Released gases gave a pressure increase in the ARC chamber that lifted the chamber lid, releasing some of the gases into the safety chamber. In one of the experiments with a version 2 cell there was a strong indication of a gas fire inside the safety chamber. Some fire scorching spots under the ARC chamber lid was observed for four of the experiments, two with version 2 cells and two with version 3 cells.

Considering the mass losses and the state of the cylinder after the experiments, the cells showed five instances of HSL 5 and one with 7. The cell with 28% mass loss was still evaluated as HSL 5 due to the state of the cell can and release of contents. Even though gas ignition was observed, this is not equivalent to HSL 6, which requires ≥1 s sustained fire.

Overall, the version 3 cells had lower mass loss and HSLs than the version 2 cells. This could be related to differences in cell versions.

As mentioned, accelerating rate calorimetry is not an initiation method. Even so, the method demonstrates the cell behaviour during a thermal event in adiabatic conditions. As for the experiments with slow heating in Chapter 4.1, the drop in cell voltage occurred before ventilation. The experiments showed that ventilation can cause the self-heating to stop, even in adiabatic conditions.

One should also keep in mind the long duration of these experiments. The time between ventilation and thermal runaway is 17–25 hours. This could cause some of the electrolyte content of the cell to dry out before thermal runaway, and this could change the thermal runaway behaviour.

Finally, the results also show that the released gases can ignite in air-filled confined spaces.

5 Discussion

Figure 5.1 summarises the results presented in this report. The main finding from the described tests is the large variation in cell behaviour. The outcome of the tests varied for all initiation methods. As an example, four identical cells with identical test set-ups exhibited hazard levels ranging from 4 to 7 (see Chapter 4.2.2). This underscores the importance of repeated testing. Abuse tests or propagation tests without repetitions can give a misleading impression of the potential hazards of the battery. Likewise, a single successful safety test example is not sufficient evidence for considering a cell as safe. Repeated testing is also necessary to reveal all possible cell behaviours. As an example, out of the nine experiments described in Chapter 4.2, the cell opened in both ends only once.

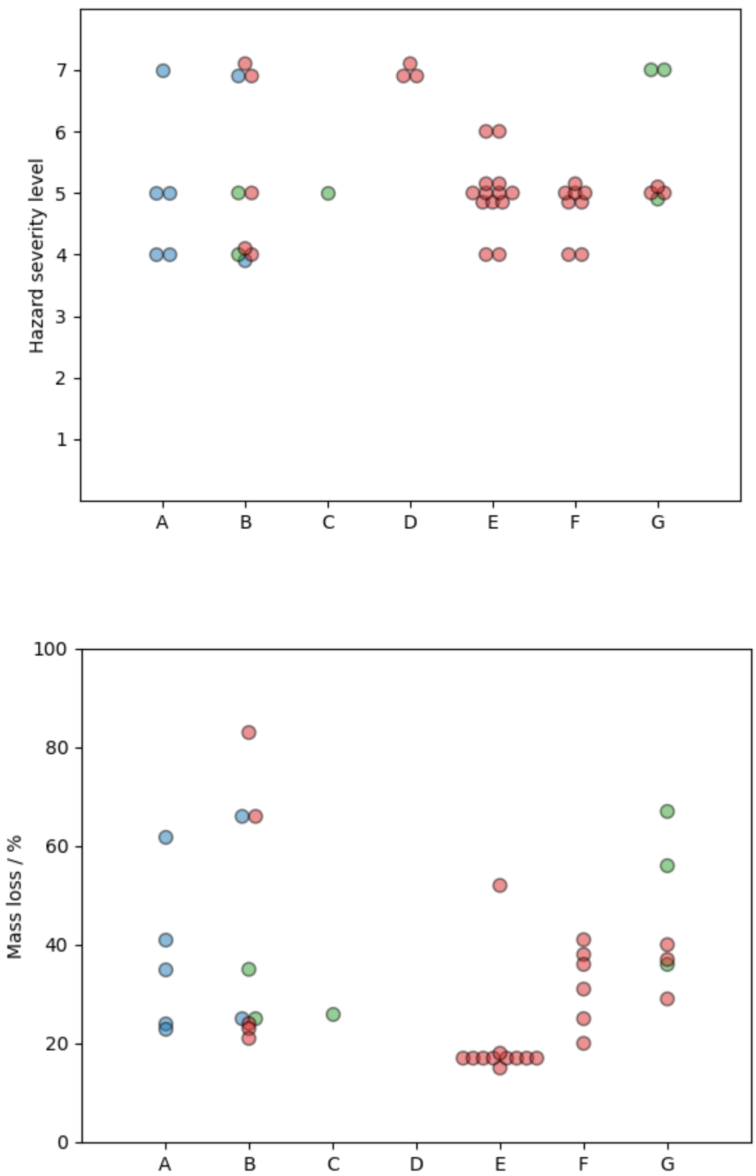

Figure 5.1 Hazard severity level (upper chart) and mass loss (lower chart) for all experiments grouped by initiation method: A: Nozzle heater 2 ºC/min. B: Nozzle heater 15 ºC/min. C: Flexible heating sheet. D: Infrared radiation heating. E: Internal heating element. F: Nail penetration. G: Accelerating rate calorimetry. Each circle represents one experiment. Circles are distributed along the x-axis when values are overlapping. Colours indicate cell version: blue – version 1, green – version 2 and red - version 3. Note that mass loss was not measured for category D.

Cell size may be a contributing factor to the observed variation in cell behaviour. In a medium sized cell, as the one studied in this report, the outcome of an abuse test can depend on where the initial internal short circuit or thermal runaway first appears [19, 20]. This location was controlled when using an internal heating element or nail penetration, but not for the other methods. If the initial short circuit appears at the opposite end of the ventilation outlet, pressure relief could be obstructed and lead to cell wall rupture. Obstruction of the ventilation outlet can also affect the mass loss during the experiment.

When comparing the results in closer detail, it is important to keep in mind that the comparison is only valid for the studied cell. The results are also valid for fully charged cells only. The results would most likely be different for another type of cell, cells of other chemistries or cells at lower SoC.

It is difficult to extract any systematic differences in cell behaviour depending on the initiation method. One exception is the method with internal heating element. For this method, there is much less variation between the experiments than for the other methods. With one exception, all cells have a mass loss between 15 and 18%. The only cell exhibiting a higher mass loss was tightly enclosed in a calorimeter (see Chapter 4.5.2). The sidewall rupture that appears when using this method apparently contributes to additional pressure relief. This averts pressure build-up and the corresponding mass ejection that was observed using the other methods. An important result is also that this method did not generate the worst-case cell behaviour observed for many of the external heating techniques, except in the mentioned case where the sidewall was tightly enclosed in the calorimeter (Chapter 4.5.2).

Figure 5.1 reveals no systematic differences between the three cell versions that were tested. For the ARC experiments, the results for version 3 cells were overall less severe than for the version 2 cells, but this can also be attributed to differences in ambient atmosphere (ref. Chapter 4.7).

Figure 5.1 shows very few instances of fire or flame (HSL 6) for the studied cell. One must keep in mind that many of the experiments in this reported were carried out in an inert atmosphere or without visual observation. This of course affects the likelihood of observing flames. For the experiments in open air, flames were observed in the experiments using a permanent ignition source (infrared radiation heater or pilot flame). Additionally, one of the externally heated cells showed burning cell material (ref. Chapter 4.24.2.2). This illustrates the flammability of the released gases and that also the cell itself can act as an ignition source. However, we never observed the characteristic jet flames that are often observed during abuse tests, especially with less thermally stable chemistries [3, 21].

However, the experiments clearly demonstrated that the flammable gases released from the cell during ventilation, can ignite. In addition, the cells ejected sparks during thermal runaway in some instances. Gases collected in confined spaces can explode when ignited by sparks, as demonstrated by some of the ARC experiments.

The results are also a reminder of the challenge with sidewall rupture for cells with rigid walls. This challenge is relevant for cylindrical cells, as these results demonstrate, but also prismatic cells. A thermal incident is more difficult to manage in a module when ventilation gases are released through arbitrary ruptures instead of their intended pressure relief devices. Module developers should be aware of this possibility, and carefully investigate if and how the module design should mitigate this.

Some results also exemplified that cell wall temperature measurements cannot be used as a reliable pre-warning parameter for thermal incidents, as pointed out by other studies [5]. In several cases, the temperature showed no changes before the thermal incident was well underway (see Chapter 4.5.2).

Figure 5.2 illustrates the difference in duration for the methods used in this report. The elapsed time before thermal runaway occurs varies over a large time scale. Nail penetration is the fastest method, giving thermal runaway in seconds only. The ARC experiments are particularly time consuming, where the cell is heated over a time span of days. Long-duration experiments can affect the cell behaviour, as discussed in Chapter 4.7.2.

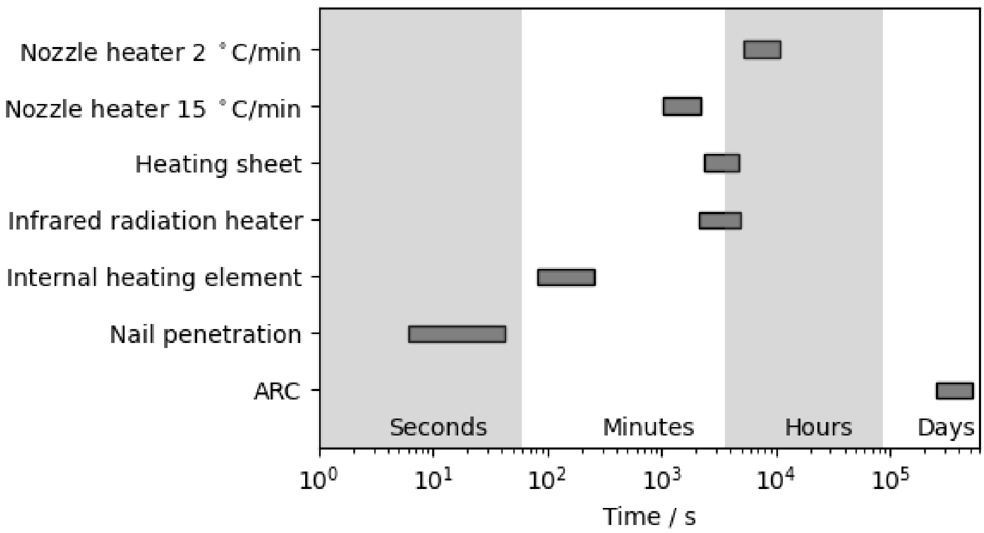

Figure 5.2 Range of durations for all experiments and methods. The duration of an experiment is here defined as the time before thermal runaway occurs, measured from start of heating or activation of abuse method.

As discussed in Chapter 4, all initiation methods have their strengths and weaknesses. Several authors have reviewed the pros and cons of available methods [6, 7, 9-11]. For the methods evaluated in this report, the method with internal heating element resembles an internal short circuit most closely. The modified cells require no additional space and can easily be used for propagation tests in modules. However, the method requires custom-made, costly cells, which can only be acquired in collaboration with the cell manufacturer. Methods with external heating can be suitable for single-cell abuse testing, but is difficult to use in propagation tests due to space constraints inside battery modules. In addition, external heating could weaken the cell wall, affecting the cell behaviour. Overall, one should seek to find a method that best resembles a realistic cell failure in an environment that best resembles the cell application.

6 Conclusion

The main findings from the described experiments can be summarized as follows: The cells revealed a large variation in cell behaviour, both for different initiation methods and for identical methods. All HSLs between 4 and 7 were observed, and mass losses ranged from 15 to 86%. This variation shows that abuse tests or propagation tests without repetitions can give a misleading impression of the potential hazards of the battery. It also shows that a single successful safety test example is not sufficient evidence for considering a cell as safe. Repeated testing is necessary to reveal all possible cell behaviours.

Out of the tested methods for initiating thermal runaway, internal heating element was the method that gave least variation in cell behaviour. This method generally did not produce the worst-case cell behaviour observed for many of the external heating methods.

Module developers and safety evaluators should be aware of the possibility for sidewall rupture when using cells with rigid walls. Ruptures can cause the ventilation gases to be released in unintended directions. The results also clearly demonstrated the flammability of the released gases and the possibility for ignition. Additionally, the results exemplified that cell wall temperature measurements cannot be regarded as a reliable pre-warning parameter for thermal incidents.

Appendix A: Hazard level comparison

Table A.1: Comparison between hazard severity levels as defined in [8] and EUCAR hazard levels defined in [12].

Hazard level	Description		Classification Criteria & Effect	
	EUCAR Hazard Level	Hazard Severity Level	EUCAR Hazard Level	Hazard Severity Level
0	No effect	No effect	No effect. No loss of functionality.	No effect. No loss of functionality.
1	Passive protection activated	Passive protection activated	No defect; no leakage; no venting, fire, or flame; no rupture; no explosion; no exothermic reaction or thermal runaway. Cell reversibly damaged. Repair of protection device needed.	No damage or hazard; reversible loss of function. Replacement or re-setting of protection device is sufficient to restore normal functionality.
2	Defect/Damage	Defect/Damage	No leakage; no venting, fire, or flame; no rupture; no explosion; no exothermic reaction or thermal runaway. Cell irreversibly damaged. Repair needed.	No hazard but damage to RESS; irreversible loss of function.

3	Leakage Δmass < 50%	Minor Leakage or Minor Vent	No venting, fire, or flame*; no rupture; no explosion. Weight loss <50% of electrolyte weight (electrolyte = solvent + salt).	Visual or audible evidence of leaking or venting. Leak without significant pooling or collection of free liquid. Venting without significant smoke or loss of particulate material. No visual obstruction of the RESS.
4	Venting Δmass > 50%	Major Leakage or Major Vent	No fire or flame*; no rupture; no explosion. Weight loss ≥50% of electrolyte weight (electrolyte = solvent + salt).	Visual evidence of leaking or venting. Leaking with significant pooling or observed free liquid. Venting with significant smoke, solvent vapor, and/or loss of particulate material. Visual obstruction of the RESS by vent gases and/or smoke. Total RESS mass loss <30%.
5	Fire or Flame	Rupture	No rupture; no explosion (i.e., no flying parts).	Loss of mechanical integrity of the RESS package, resulting in release of contents. The kinetic energy of released material is not sufficient to cause physical damage external to the RESS. Rupture may be the result of a RESS thermal runaway (but not necessarily). Total RESS mass loss 30–55%

6	Rupture	Fire or Flame	No explosion, but flying parts of the active mass.	Ignition and sustained combustion of flammable gas or liquid (≥1 s sustained fire). Sparks or incandescent material is not considered a fire or a flame.
7	Explosion	Energetic Failure	Explosion (i.e., disintegration of the cell).	Fast release of energy sufficient to cause pressure waves (slower than the speed of sound) and/or projectiles that may cause considerable structural and/or bodily damage, depending on the size of the RESS. The kinetic energy of flying debris from the RESS may be sufficient to cause damage as well. Total RESS mass loss ≥ 55%.
*The presence of flame requires the presence of an ignition source in combination with fuel and oxidizer in concentrations that will support combustion. A fire or flame will not be observed if any of these elements are absent. For this reason, we recommend that a spark source be use during tests that are likely to result in venting of cell(s). We believe that “credible abuse environments” would likely include a spark source. Thus, if a spark source were added to the test configuration and the gas or liquid expelled from the cell was flammable, the test article would quickly progress from level 3 or level 4 to level 5.				

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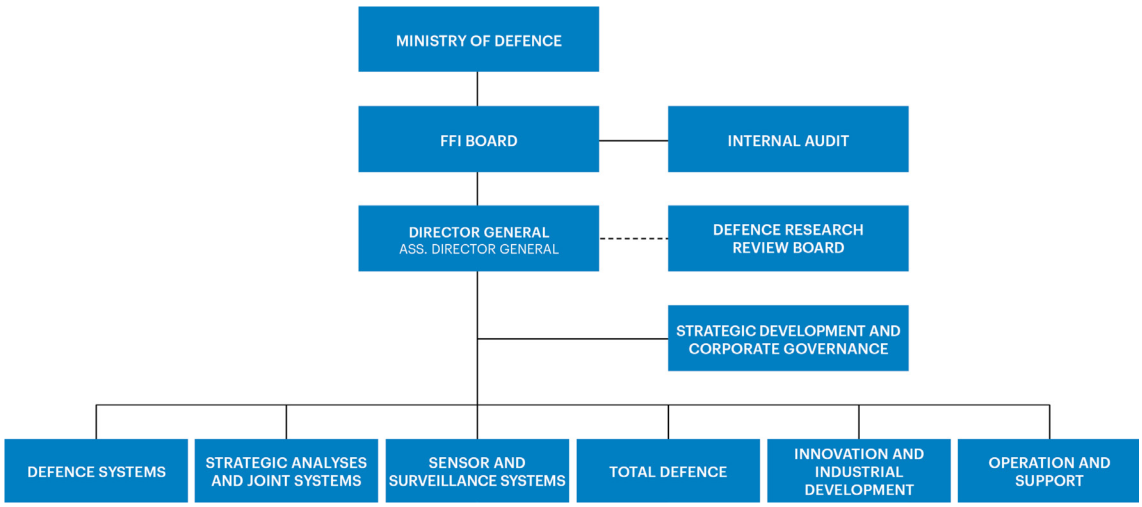

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