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On thermal safety characteristics of rechargeable alkaline batteries based on zinc and manganese dioxide

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ABSTRACT

As lithium-ion technology's exhibits inherent issues with safety due to thermal runaway, a sustainable and cheaper alternative has been proposed in this work: the rechargeable alkaline battery chemistry. However, so far, the postulated safety of the new battery chemistry has not been demonstrated adequately. Therefore, a safety study is being carried out for rechargeable alkaline battery cells. This Short Communication paper is the first report on the thermal safety of $Zn\text{-}MnO_2$ CR2032 rechargeable alkaline battery coin cells. 100 % charged coin cells were tested under thermal abuse conditions in a gravity-convection furnace to quantify the temperature at which the cell would go into thermal runaway. Morphological characterisation of pristine and tested cells was performed via laboratory-based X-ray computed microtomography. The onset temperature to thermal runaway for the rechargeable alkaline battery cells was found to be in the range of 290–380 °C, much higher than that reported in the literature for lithium-ion cells (150–200 °C) of similar capacity and geometry. These results emphasise that rechargeable alkaline battery technology has improved thermal stability compared to lithium-ion technology. Lastly, morphological analyses highlighted the variations of cell geometry brought about by thermal testing.

1. Introduction

The lithium-ion battery (LIB) technology is the state-of-the-art for automotive, electrical and consumer electronics (Xiong and Shen, 2018; Huang et al., 2024). Notwithstanding their widespread use, it is well known that LIBs are potentially hazardous if subjected to abuse conditions and a large number of catastrophic fire incidents in various industries have been reported (Kong et al., 2023; Liu et al., 2022). Together with sustainability issues, safety is a key driving force for the development of alternative technologies; sodium-ion batteries follow the sample principles of LIB technology; the advantage of sodium-ion batteries (SIB) is their comparatively large abundance. SIB safety issues are similar to those of LIB technology (Robinson et al., 2018), as SIB can also result in thermal runaway (Cui et al., 2022; Yue et al., 2024). Similarly, less-developed potassium and calcium-ion batteries also present safety concerns as they exhibit similar mechanisms that lead to thermal runaway (Zhang et al., 2023). LIB and SIB technologies for grid storage are also expensive as they cost around 200 USD/kWh; alkaline zinc-manganese oxide ($Zn\text{-}MnO_2$) batteries are an alternative, safer, low-cost technology (<100 USD/kWh) and could be used for grid storage systems (Lim et al., 2021), or – after appropriate technological developments – the automotive industry. In principle, rechargeable Alkaline Batteries (RABs) may ensure intrinsic safety.

RABs had been actively investigated in the '80 s and '90s but never reached the full industrial level because the cathode and anode rechargeability issue had not been fully understood and solved. Recently, interest in this chemistry has been revived thanks to the achievement of new insight into the reversible electrochemical behaviour of Zn and MnO_2 in a close-knit group of research institutions, among which the groups of the Authors of this work (Rossi et al., 2022a; Bozzini et al., 2023; Salman et al., 2023). The RAB is one of the few post-lithium technologies being currently developed that combine the following features: (i) power and energy density appropriate for automotive applications; (ii) intrinsic safety, based on the absence of

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flammable or high-energy electrode materials and use of an aqueous electrolyte and (iii) use of exclusively non-toxic and sustainable raw materials. Moreover, if the electrode materials are formulated in the form of slurries that can be tape-cast, the fabrication lines of state-of-the-art LIBs gigafactories can be straightforwardly converted for the production of RABs. Thus, after the fabrication of the first RAB prototypes realised in the authors' laboratories, the quantification of their thermal stability behaviour, proposed for the first time in this paper, is a crucial step towards industrialisation.

Several steps and phenomena merit experimental investigation to determine the safety of battery technology in various applications. The first assessment for a battery cell is its thermal stability, at which point it undergoes thermal degradation and thermal runaway. Thermal degradation studies use Acceleratory Rate Calorimetry (ARC) for lithium-ion batteries, as reported in Yue et al. (2024), Zhang et al. (2024), Liu et al. (2023). Yue et al. (2024) Also, the cell components, such as electrolytes, are often studied via Differential Scanning Calorimetry (DSC) or Thermogravimetric Analysis (TGA) (Liu et al., 2023; Zhou et al., 2024; Wang et al., 2024; Yang et al., 2022). Another inexpensive way to investigate thermal degradation is by performing thermal abuse tests in traditional convective furnaces (Abada et al., 2018; Kim et al., 2007). The subsequent phenomenon after thermal runaway is venting along with ejecta; if early ignition of the gases occurs, then jet flames (momentum-dominated) and buoyant-dominated flames will take place. However, deflagration is the most likely scenario if the gases generated are mixed with the oxidiser in the air and delayed ignition occurs. Therefore, it is essential to investigate battery technology's fire and explosion behaviour at the cell and application levels and their effect on closed spaces or nearby infrastructure (Huang et al., 2024; Kong et al., 2023; Liu et al., 2022; Bai et al., 2024).

In RAB technology, various electrolytes are proposed; however, some types of electrolytes present several challenges (Meng et al., 2024). Aqueous electrolytes might lead to battery failure as the cathode material can dissolve at the electrolyte-electrode interphase (Salman et al., 2023; Zhao et al., 2019; Ji, 2019), and the anode can undergo shape changes limiting cyclability (Bozzini et al., 2023; Kazemian et al., 2022; Rossi et al., 2022b; Emanuele et al., 2024). Moreover, recharge abuse can lead to hydrogen gas and zinc corrosion formation, resulting in safety hazards (Zhang et al., 2021). Therefore, it is highly relevant to study the thermal stability of RAB cells under an array of scenarios to find potential hazards and demonstrate their safety. To the authors' knowledge, no literature studies are reporting the thermal runaway of RAB cells; only a webpage post reported that the RAB technology is safe, and no thermal runaway was observed when the cell was subjected to the standard UL 1973 test (UEP,). The current preliminary study studied their thermal stability in a furnace using thermal abuse tests. In addition, x-ray computed tomography was used to characterise the cells prior to and after thermal runaway; the technique has been used before to provide insights into half or full cells *in situ* (Bozzini et al., 2023, 2020) and *post-mortem* (Nagourney et al., 2021).

2. Materials and methods

2.1. The RAB cell

The RABs tested in this project were 100 % fully charged CR 2032, with zinc anodes and manganese dioxide cathodes (Kazemian et al., 2022), ZnO with α-MnO_2 and obtained free-standing pellets. The anodes were Zn discs, 12 mm in diameter, punched from 250 μm thick foil (Goodfellow). The cell capacity is 50 mA h. In this study, we employed six cells in the as-fabricated state. The cells were equipped with a K-type thermocouple with wires 0.52 mm in diameter: the two thermocouple ends were fixed with a Kapton tape to the positive and negative terminals of the battery. In addition, four commercial VARTA primary 100 % fully charged CR203 LIB cells (Li-Manganese dioxide/Organic Electrolyte - 3 V 220 mA h) were also tested as the control group.

Table 1
Experimental battery thermal test matrix.

Cell Number	Objective
RAB1, RAB2, and RAB 3	The cells are subjected to thermal abuse for two hours up to 200 °C and then are taken to be studied for 3D microstructural characterisation.
RAB4, RAB5, RAB6, RAB7, RAB8, and RAB6	The cells are subjected to thermal abuse in a preheat furnace at 300 °C until thermal runaway is attained.
LIB1, LIB2, LIB3, and LIB4	The control group were tested at 200 °C.

2.2. Thermal abuse testing

Thermal abuse testing of the RAB cells was performed using two protocols, detailed in Table 1. Specifically, three cells were tested in the furnace for a specific time, removed before having reached thermal runaway, and subjected to x-ray XmCT. The second group of three RAB cells along with the control group cells was tested to determine the onset of thermal runaway. The RAB cells were tested in a small gravity-convective BOSIO furnace with 1 °C temperature sensitivity, and control group cells were tested in a TTC256 Temperature test chamber from CELAB with 0.5 °C temperature sensitivity. Following literature methods (Guo et al., 2010; Hatchard et al., 2001), the furnace was preheated to the set temperature. Then, the cell under test was rapidly introduced in the furnace with two attached thermocouples (on the positive and negative coin cell sides). A third thermocouple measured the temperature of the furnace air.

2.3. 3D microstructural characterisation

The XmCT technique has been applied to pristine and abused samples to obtain, by a non-destructive approach, the 3D morphological characterisation of the batteries. The applied multiscale imaging method allowed us both to analyse the whole cell and selected volumes of interest. We employed a Zeiss Xradia MicroXCT-400 instrument based at ZAG (Ljubljana, Slovenia). The instrument is equipped with a microfocus X-ray source operating in the voltage range of 40–150 kV and a CCD camera coupled with a multi-lens system, allowing work with spatial resolution from ca. 1 to a few tens of μm on samples with different sizes. The reconstruction of XmCT scans allowed the detection of morphological features of interest (pores, grains, layers and cracks) related to the manufacturing process (pristine cells) or the ageing of the battery (cells submitted to abuse tests) up to thermal runaway or terminating the test at intermediate states. XmCT scans were acquired with the following settings: Voltage = 139 kV, Power = 10 W, exposure time/projection = 2 s, 1600 projections acquired over 360°; the instrument was set to acquire images at two different spatial resolutions, corresponding to effective pixel sizes of 7.95 and 20.6 μm. The tomographic reconstruction was performed by using the Zeiss commercial software provided with the XmCT instrument. The visualisation of bi-dimensional (2D) virtual sections of the sample was obtained by the open-source software Fiji (Bozzini et al., 2023), while the 3D visualisation of the data, through volume rendering procedures, was obtained with the commercial software Dragonfly 2022.2 (ORS, Canada).

3. Results and discussion

In this chapter, the results of the thermal abuse experiments and the 3D morphological characterisation will be presented and discussed in their respective sections. In addition, the current thermal abuse results are compared with the literature for LIB.

3.1. Thermal abuse

As put forward above, thermal abuse experiments were aimed at determining the thermal damaging onset temperature and the thermal

Table 2
Summary of the thermal damaging onset temperature to thermal runaway for the RAB cells obtained during thermal-abuse testing at var 300 °C.

	RAB cell number						LIB cell number			
	RAB4	RAB5	RAB6	RAB7	RAB8	RAB9	LIB1	LIB2	LIB3	LIB4
Onset temperature prior to thermal runaway [°C]	291	‡	380	376	314	337	156	157	156	155

‡ No thermal runaway observed.

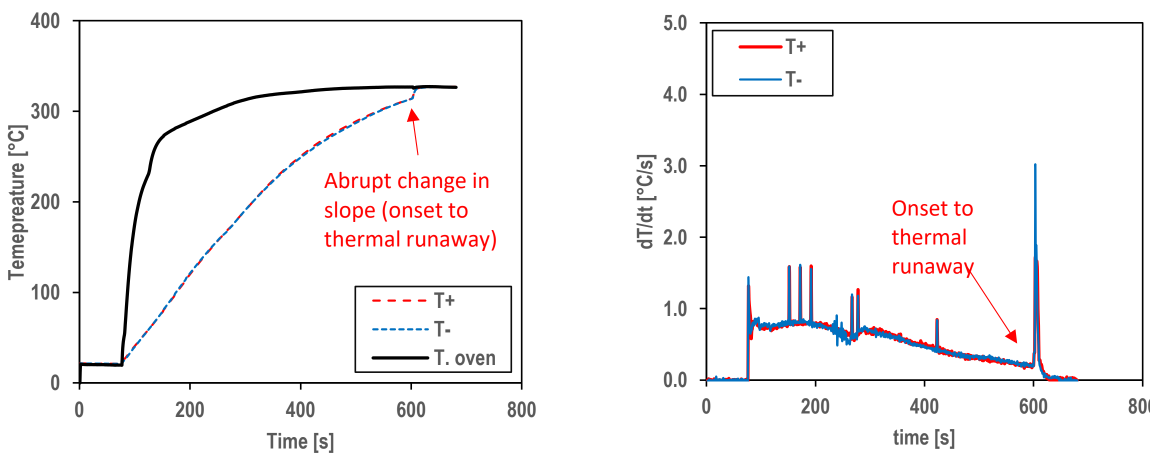

Fig. 1. Left panel: temperature readings for RAB8 and the furnace during testing at 300 °C. Right panel: the corresponding derivative of the temperature profile (Zhao et al., 2022).

runaway temperature of the RAB cells. The onset temperatures prior to thermal runaway results for the six RAB cells and the control cells (LIB) are listed in Table 2. The onset temperatures vary in the relatively wide range of 291–380 °C, witnessing the complexity of thermal damaging mechanisms, which, nevertheless, occurs at notably higher temperatures than other battery chemistries (Rossi et al., 2022b; Emanuele et al., 2024; Zhang et al., 2021; UEP; Bozzini et al., 2020; Nagourney et al., 2021; Guo et al., 2010). The comments in Fig. 1 provide details on the occurrence of thermal runaway. One can notice that thermal runaway typically occurs at temperatures in excess of 300 °C. Similar experiments with primary LIB commercial coin cells results show thermal runaway at temperatures just above 150 °C.

A typical complete thermal measurement is reported for reference in Fig. 1. The left panel shows the thermocouple readings for the RAB8 cell, corresponding to the positive and negative sides, together with the thermocouple reading for the furnace temperature. The right panel displays the corresponding temperature derivative, highlighting subtle trend changes better. As expected, the furnace temperature profile is characteristic of convective heating, while the thermocouples measuring the cell exhibit a lower heating rate since the battery behaves as a heat sink, controlled by the thermal resistance of stainless steel casing (thermal conductivity of 15 W/mK) and the heat inertia of the battery components. During the experiment, the RAB was found to reach thermal equilibrium with the oven through an abrupt temperature increase, denoting the onset of battery thermal damage, preceding *bona fide* thermal runaway.

It is instructive to compare our original RAB cells and the control group cells' thermal response results with similar ones obtained for other battery technologies. Three 85 mA h CR2032 coin cells at 100 % SOC ($LiNi_{0.6}Mn_{0.2}Co_{0.2}O_2$) exhibited onset temperatures ranging from 157 to 164 °C (Zhao et al., 2022). LIBs have low onset temperatures, typically 150–200 °C. Our measurements show that RABs show notably larger runaway temperatures: 290–380 °C.

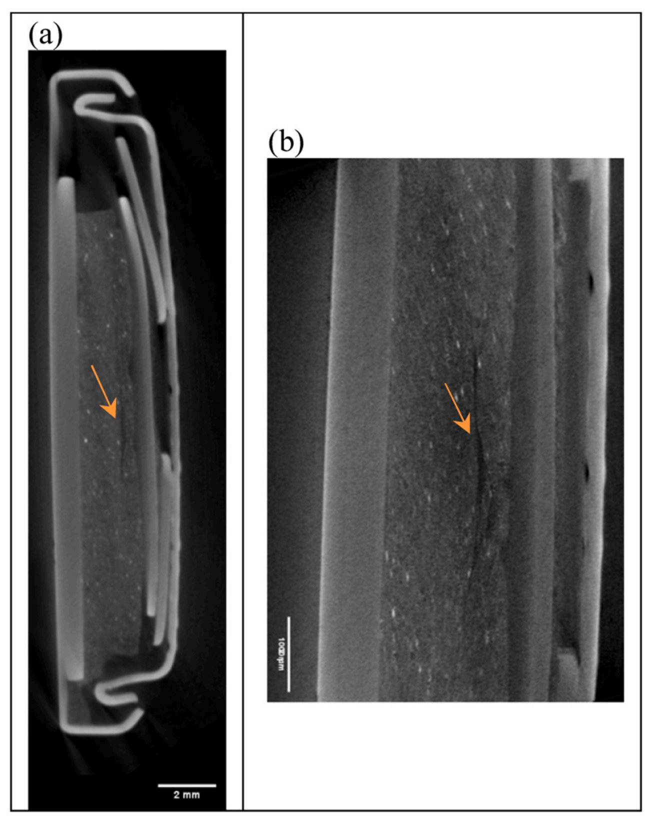

Fig. 2. Two representative transversal virtual sections of the fully charged, pristine RAB at different spatial resolutions. Tomographic reconstruction was performed with isotropic voxel sizes of (a) 20.6 µm and (b) 7.95 µm.

3.2. 3D morphological characterisation

This work provides experimental evidence for mechanism

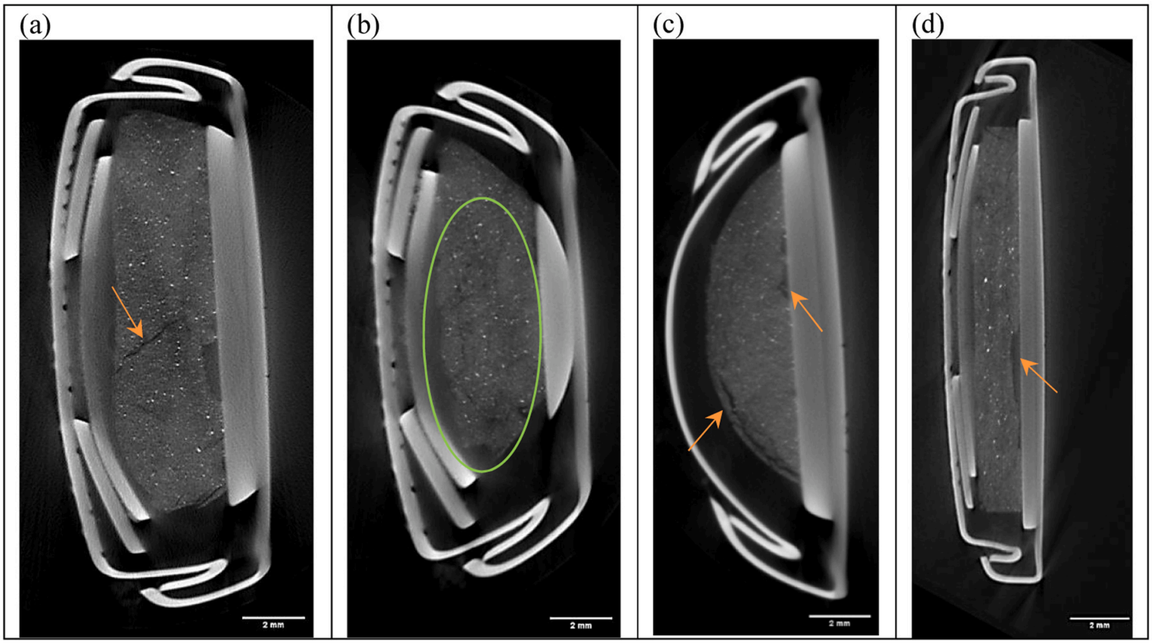

Fig. 3. Typical virtual axial (a–c) and transversal (d) sections of the RAB after thermal abuse for 2 h at 200° C. Tomographic reconstruction performed with an isotropic voxel size of 20.6 μm.

determination with 3D imaging. Specifically, three RAB cells (RAB1, RAB2, and RAB3) were thermally abused for two hours at 200 °C, i.e. well below the thermal runaway threshold. Tomographs of pristine and thermally abused cells are compared.

In Fig. 2, we report transversal slices recorded at different spatial resolutions of the fully charged, pristine RAB battery (the remaining images for the other RAB batteries will be placed in supplementary material). In addition to the auxiliary stainless steel components of the cell (casing (a), spacers (b) and conical spring (c), the cathode (d), separator (e) and anode (f) can be observed. The Zn metal foil anode (f) is perfectly homogeneous at the scale investigated, while the cathode (d) exhibits a granular morphology, characterised by highly absorbing MnO_2 agglomerates (white contrast) embedded in the carbon-based matrix. Some cracks (g) form in the cathode material due to the cell sealing step in the fabrication process.

In Fig. 3 several virtual sections of sample RAB3 are shown. Regions of interest have been highlighted with orange arrows and a green circle. In these regions cracks and a clear indication of the detachment of the active material from the metallic support are visible.

It is not clear what mechanisms drove the morphological change, but it could be hypothesised that those changes might have been induced by thermal abuse due to a mechanical kind of damage that can be straightforwardly described as the compression failure of the less elastic and brittler cathode composite, resulting from differential thermal expansion, accompanied by the evaporation of the minimal amount of aqueous electrolyte. No indications could be spotted of the chemical reaction of MnO_2 or carbon and the decomposition of the organic binder.

4. Conclusions

Under thermal abuse, the Rechargeable Alkaline Battery cells investigated in this study exhibited an average onset temperature to thermal runaway range typically above 300°C, well in excess of temperatures typical of Lithium-ion batteries (150–200 °C). Combining thermal tests with 3D X-ray microscopy imaging, we could correlate the thermal response to the structural modifications of the internal cell components. We detected cracking of the cathode and detachment of the cathode from the current collector, together with the bulging of the casing, denoting merely mechanical damage due to differential thermal expansion of the constrained, more brittle cathodic material.

The obtained results, for the first time, qualify the thermal abuse response of RABs. On the one hand, we proved experimentally that the RAB coin cells are substantially more thermally stable, and on the other hand, we have gained positive evidence that can guide further studies on damaging mechanisms. In particular, in our group, research is continuing in the following directions: (i) fabrication of prismatic RABs for stationary storage applications; (ii) deepening of the understanding of thermal degradation and safety behaviour based on acceleratory rate calorimetry (ARC) and DSC; (iii) computational modelling.

CRediT authorship contribution statement

Emanuele Marini: Resources, Methodology, Investigation, Funding acquisition. **Benedetto Bozzini:** Writing – review & editing, Writing – original draft, Supervision, Resources, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Ulises Rojas-Alva:** Writing – review & editing, Writing – original draft, Visualization, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Alenka Mauko Pranjić:** Writing – original draft, Resources. **Lucia Mancini:** Writing – review & editing, Writing – original draft, Visualization, Software, Resources, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.psep.2025.107175.

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