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Cycle life of lithium ion batteries after flash cryogenic freezing

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ABSTRACT

Growing global sales of electric vehicles (EVs) are raising concerns about the reverse logistics challenge of transporting damaged, defective and waste lithium ion battery (LIB) packs. The European Union Battery Directive stipulates that 50% of LIBs must be recycled and EV manufacturers are responsible for collection, treatment and recycling. The International Carriage of Dangerous Goods by Road requirement to transport damaged or defective LIB packs in approved explosion proof steel containers imposes expensive certification. Further, the physical weight and volume of LIB packaging increases transport costs of damaged or defective packs as part of a complete recycling or repurposing strategy. Cryogenic flash freezing (CFF) removes the possibility of thermal runaway in LIBs even in extreme abuse conditions. Meaning damaged or defective LIBs may be transported safely whilst cryogenically frozen. Herein, LIBs are cycled until 20% capacity fade to establish that CFF does not affect electrical performance (energy capacity and impedance) during ageing. This is demonstrated on two different cell chemistries and form factors. The potential to remanufacture or reuse cells/modules subjected to CFF supports circular economy principles through extending useful life and reducing raw material usage. Thereby improving the environmental sustainability of transitioning from internal combustion engines to EVs.

1. Introduction

Market adoption of hybrid electric vehicles, plug-in hybrid electric vehicles and battery electric vehicles containing lithium ion batteries (LIB) continue to accelerate; for example over 2 million plug-in vehicles were sold worldwide in 2018, adding to the 5 million already on the road [1]. Despite improvements in battery durability [2], ageing mechanisms, such as solid electrolyte interphase layer growth [3–6], cause LIBs to eventually no longer store sufficient energy capacity for their original automotive application [7–9]. End of life (EoL) for automotive applications is commonly defined as when the battery capacity has reduced to 80% capacity when compared to new [10] or when the internal impedance has doubled [11]. In-field failures or road traffic accidents which damage the battery pack also result in premature EoL for the LIB. The Circular Energy Storage, a London based research and consulting group, estimates the global EoL LIB market to be currently worth $1.3 billion, with the LIB second life market expected to reach $4.2 billion whereas the recycling market is predicted to grow to $3.5 billion by 2025 [12].

Despite the growing size of the EoL markets, protocols and procedures for LIB EoL, such as recycling, remanufacturing and re-use, e.g. [13], are not well established [14,15]. As discussed in [16], there are a number of barriers opposing the reverse logistics required to support second life applications. The current legislation such as the UK Batteries Directive [17] and the European agreement concerning the International Carriage of Dangerous Goods by Road (ADR) [18] make it challenging to transport damaged or defective batteries, which is a prerequisite for LIB second life. Further, higher costs are incurred and only specialised logistics firms are typically able to provide this service. Unless these LIBs can be shown to be safe, which is defined as not being able to explode, vent dangerous gases, catch fire, or go into thermal runaway (as per the requirements of ADR [18]), they must be transported in accordance with transport category 0 as per ADR SP376 [18]. Practically, this means using specialised transport service providers, e.g. using explosion proof steel containers, which cost circa €10,000 for a typical Tesla sized pack and a further circa €10,000 for the UN accreditation [19]. In this paper, the authors have not established if cryogenic freezing will be more cost effective since the aim of this body of work is to first establish if the process of making LIBs safe through cryogenic freezing is at all viable from a LIB performance point of view.

Abbreviations: ADR, European Agreement concerning the International Carriage of Dangerous Goods by Road regulations; CC, constant current; CV, constant voltage; DK, Dow Kokam; EOL, End of life; EV, Electric vehicle; HPPC, Hybrid Pulse Power Characterisation; HVM, High Value Manufacturing; LIB, Lithium ion battery; NCA, nickel cobalt aluminium oxide; NMC, nickel manganese cobalt oxide; SOC, state of charge

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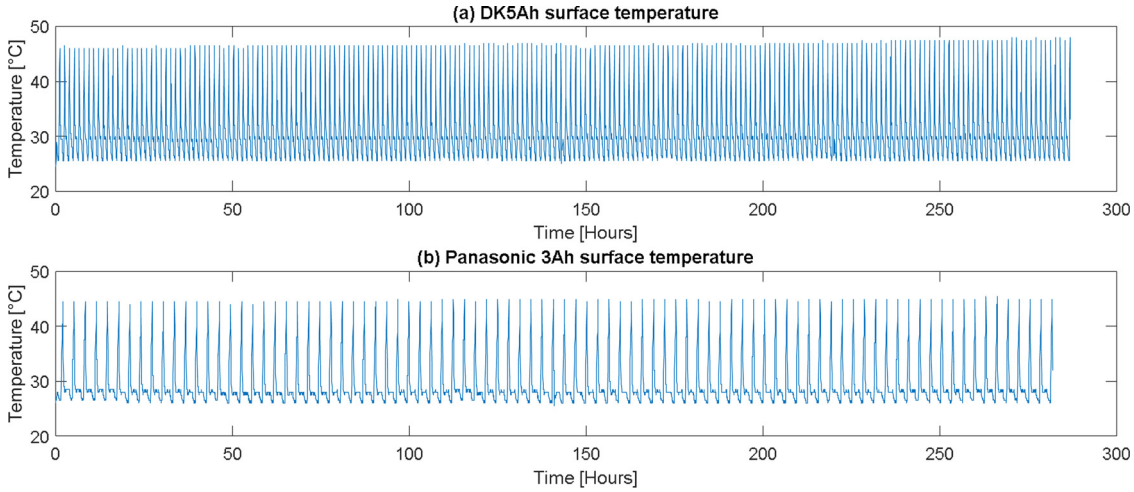

Fig. 1. Cell surface temperature of (a) DK 5 Ah cells and (b) Pan 3Ah during cycling.

Once proof-of-concept has been established, as discussed in Section 3.3, the authors recommend a full economic assessment of the methodology that will further underpin commercialisation and adoption by industry.

In reality, it is normally impractical to establish whether a damaged or defective LIB is safe because the LIB is not conforming to the type tested according to UN38.3 [20]. Additional testing would be required to ensure the LIB is safe, which is not practical to perform at a road traffic accident site. Effectively, LIB packs with no or only relatively minor damage will end up discarded because it is not economically viable to transport them to a battery remanufacturing or re-use facility. A large LIB, which can contain thousands of individual lithium-ion cells, can be rendered damaged or defective by a proportion of the cells being damaged or defective or by ancillary failures, e.g. a failure of the BMS to report battery status. Therefore, depending on the failure mode, most of the cells in a damaged or defective pack could still be reusable. In addition to LIBs being transported from first usage in motor vehicles to a battery treatment centre for remanufacturing, re-use or second life applications, the research is also relevant to new batteries being delivered to a car manufacturers.

The authors have recently demonstrated that lithium ion cells are safe when they are cryogenically frozen [21], which should comply with the requirements of ADR SP376 [18] and permit their transport without the use of expensive explosion proof steel containers. Furthermore, results presented in [21] reveal that the cryogenic freezing has little to no-effect on the electrical performance (energy capacity and internal resistance) of the two different cell chemistries and form factors tested. A recent study confirmed this finding on 18,650 lithium-ion cells after a 14-day cryogenic freezing period [22]. This solution would therefore facilitate the potential repair or remanufacturing of individual cells and modules, prolonging the useful life, as well as support second life applications for LIBs [23]; improving the environmental sustainability of EVs. In this paper, for the first time, the authors present the capacity degradation of cryogenically frozen LiBs cycled over several months compared to LiBs that was not frozen.

The effects of flash cryogenic freezing on the life cycle of LIBs has not yet been reported in the literature. This initial study therefore aims to establish if cells that have been cryogenically frozen age and degrade normally when cycled over several months, in order to establish if there are any effects of the life expectancy of LIBs.

2. Experimental method

The experimental method consists of cycling lithium ion cells in an Espec thermal chamber set to 25 °C that had been cryogenically frozen prior to the experiment and comparing the results against a control group of cells that have not been subject to the same frozen conditions. The full detailed methodology for cryogenically freezing the cells and evaluating the electrical performance of the cells using the energy capacity and Hybrid Pulse Power Characterisation (HPPC) measurement is described in full detail in [21]. Briefly, the HPPC test consisted of 10 pulses (5 charge and 5 discharge with current values of 2, 4, 6, 8, and 10 A) applied at 90%, 50% and 20% SOC at 25 °C after leaving the cells to equilibrate electrochemically and thermally for three hours as per the method defined in IEC-62660 [24]. This short communication only describes the cycling performed. For consistency, the same cell selection is used as in our previous study [21]: i.e. Dow Kokam (DK) 5 Ah 100 x 106 mm nickel manganese cobalt oxide (NMC) pouch and Panasonic 3 Ah 18,650 nickel cobalt aluminium oxide (NCA) cylindrical cells. Although the DK 5 Ah is not a cell currently used in EV traction batteries, NMC is the most popular of the chemistries currently used by EV manufacturers, such as Kia, Hyundai, BMW and Mercedes-Benz.".

Six DK 5 Ah cells were used and divided into two groups, where n is the number of cells in each group.

1 DK01: cryogenically frozen before cycling (n = 3)
2 DK02: control (n = 3)

Both groups were cycled 105 times before energy capacity measurements are performed, and 210 times before HPPC measurements. One cycle consists of fully discharging the cells at constant current of 4C (20A) to their lower voltage threshold (2.7 V). The cells were rested for 10 min prior to being fully charged using a constant current (CC) of 1C (5A) to the upper voltage defined by the manufacturer (4.2 V) followed by a constant voltage (CV) phase until the current reduced to 0.1A. There is another 10 min rest before the next cycle begins. In order to maintain safety throughout the experiments, a maximum surface temperature of 60 °C was established. After the 150th cycle, since the surface temperatures of the cells were below the threshold temperature, as shown in Fig. 1(a), the cycle rates were increased to the maximum C-rates on the manufacturer's datasheet (2C charge and 5C discharge) for both groups for the remaining of the experiment. A paired t-test[1] with p-value = 0.05 for each measurement is performed in order to establish if there is any statistically significant difference between the two battery groups: DK01 and DK02.

Likewise, six Panasonic 18,650 cells were also divided into two groups, where n is the number of cells in each group.

1 PAN01: cryogenically frozen before cycling (n = 3)
2 PAN02: control (n = 3)

Both groups are cycled (0.5C charge, 1C discharge) 45 times before energy capacity measurements are performed, and 90 times before HPPC measurements. One cycle consists of fully discharging the cells at constant current of 1C (3A) to their lower voltage threshold (2.5 V). The cells were rested for 10 min prior to being fully charged using a constant current (CC) of 0.5C (1.5A) to the upper voltage defined by the manufacturer (4.2 V) followed by a constant voltage (CV) phase until the current reduced to 0.1A. There is another 10 min rest before the next cycle begins. After the 90th cycle, since the surface temperatures of the cells were below 50 °C, as shown in Fig. 1(b), the cycle rates were increased to the maximum C-rates on the manufacturer's datasheet (0.5C charge and 2C discharge) for both groups for the remaining of the experiment (900 cycles). A paired *t*-test with p-value = 0.05 for each measurement is performed in order to establish if there is any statistically significant difference between the two groups, i.e. the null hypothesis that both groups have the same mean.

3. Results and discussion

3.1. Energy capacity

The DK 5Ah and Panasonic 3Ah capacity average measurements ± standard error are presented in Fig. 2. The standard error is the sample standard deviation divided by the square of the root of the sample size.

Fig. 2(a) shows that in the beginning (first eight data points) there is very little variation between the energy capacities of the DK5 Ah cells that were cryogenically frozen (DK01 – blue trace) and the control group (DK02 – red trace). After approximately 600 cycles, the two groups appear to diverge, with the control group having lower average capacity than the cryogenically frozen one. The DK 5 Ah capacity *t*-test results are summarised in Table 1, which are all > 0.05, therefore the difference is not statistically significant. The results obtained support accepting the null hypothesis that both groups have the same means. This finding should be confirmed with a larger sample size.

Similarly, Fig. 2(b) shows that the Panasonic 3 Ah capacity measurements that are very similar for the group that were cryogenically frozen (DK01 – blue trace) and the control group (DK02 – red trace). Table 2 summarises the Panasonic 3 Ah capacity *t*-test results, with a confidence level > 0.05. As for the previous case, therefore we accept the null hypothesis and conclude that both groups have the same mean value of energy capacity. It can therefore be concluded that for both the DK 5 Ah and the Panasonic 3 Ah, the flash cryogenic freezing does not affect the cycle life.

Table 1
DK 5 Ah capacity *t*-test results.

Cycles	*t*-test	Cycles	*t*-test	Cycles	*t*-test	Cycles	*t*-test	Cycles	*t*-test
0	0.19	469	1.00	1309	0.34	1834	0.69	2359	0.26
75	0.33	574	0.84	1414	0.38	1939	0.71	2464	0.27
150	0.57	679	0.94	1519	0.36	2044	0.67		
259	0.58	1099	0.45	1624	0.38	2149	0.40		
364	0.78	1204	0.40	1729	0.38	2254	0.38		

Table 2
Panasonic 3 Ah capacity *t*-test results.

Cycles	*t*-test	Cycles	*t*-test	Cycles	*t*-test	Cycles	*t*-test	Cycles	*t*-test
0	0.64	180	0.54	360	0.77	630	0.75	810	0.62
45	1.00	225	0.64	405	0.58	675	0.84	855	0.61
90	0.28	270	0.83	540	0.57	720	0.63	900	0.28
135	0.12	315	0.86	585	0.64	765	0.22		

3.1.1. Energy capacity recovery during cycling

Fig. 2(a) shows that the energy capacities of the DK5 Ah cells monotonically decrease as the cells are electrically and thermally cycled. This is a well-documented phenomenon within the literature: the energy capacity of LIBs deteriorates due to ageing mechanisms such as solid electrolyte interphase layer growth and structural changes to the electrode [3–5]. However, Fig. 2(b) shows that the Panasonic 3 Ah capacity measurements increase at four points in the cycling process, i.e. the average capacity measurements at 360, 540, 810, and 900 cycles are greater than the corresponding previous measurement (all six cells exhibit this behaviour). This is unusual since the additional cycles would normally reduce the overall cell capacity. Each of these instances occurred after the cycling was paused, e.g. after the capacity measurement at 765 cycles, the testing did not resume immediately due to facilities constraints. All tests were conducted with the same cyclers that was calibrated to the manufacturer's recommendations. The cells were stored for 2 weeks before they were cycled and the capacity measurement at 810 cycles was performed. It is interesting to note that the DK5 Ah were mounted on the same jig and their cycling was also paused four times (at 784, 994, 1624, and 1834 cycles) like the Panasonic 3 Ah cells, however they do not show this apparent capacity increase (see Fig. 2(a)). This phenomenon was only observed in aged Panasonic 3 Ah cells, which are comprised of NCA with a LiC_6 (graphite) anode. Conversely, the internal chemistry of the DK5 Ah cell is NMC. This reversible capacity effect has been investigated in automotive 25 Ah prismatic NMC cells and attributed to anode overhang [25]. However, Epding et al. 2019 [26] refutes anode overhang as the

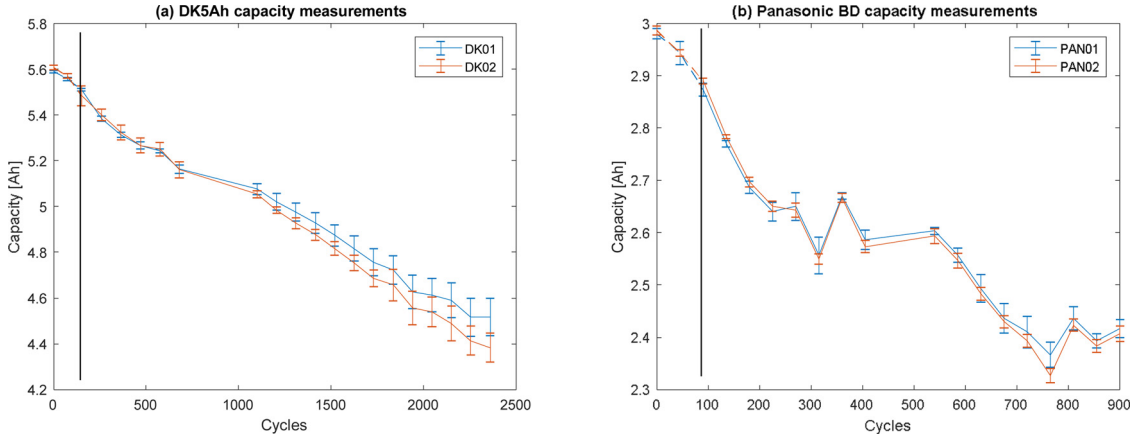

Fig. 2. Capacity measurement of (a) DK 5 Ah cells and (b) Pan 3Ah after cycling (control group in red). The lines indicate when the cycling rates were increased. The freezing was done prior to cycling. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

mechanism responsible for the phenomenon since a capacity increase is measured in cells stored at 100% state of charge (SOC) during the rest period, and suggests it is linked to the occurrence of lithium plating. Regardless of the mechanism, the experimental method was robust: energy capacity measurements were performed as per the recognised protocols defined in IEC-62660 [24], which includes long relaxation times, i.e. 720 min for the thermal stabilisation before fully electrically charging the cells and 180 min prior to the cells being discharged to measure their capacity. Further testing revealed that once the cells were sufficiently aged (approximately 300 cycles), the capacity measurements would creep up for ˜175 h. Further work is being undertaken to investigate the very slow electrochemical effects that influence capacity measurements in aged Panasonic 3 Ah. However, since the cryogenically frozen cells and the control both display this phenomenon, it does not detract from the findings reported here, that is to say the flash cryogenic freezing does not adversely affect the cycle life of DK 5 Ah and Panasonic 3 Ah cells.

3.2. Internal resistance

The DK 5 Ah and Panasonic 3 Ah internal resistance average measurements ± standard error are presented in Fig. 3.

Fig. 3(a)–(c) show that in the beginning (first four data points) there is very little variation between the internal resistances of the DK5 Ah cells that were cryogenically frozen (DK01 – blue trace) and the control group (DK02 – red trace). After approximately 600 cycles, the two groups appear to diverge, with the control group having higher internal resistance than the cryogenically frozen one. The DK 5 Ah internal resistance *t*-test results are summarised in Table 3, for a confidence interval of 0.05, therefore the difference is not statistically significant. We accept the null hypothesis that both groups have the same means.

Similarly Fig. 3(d)–(f) show that the Panasonic 3 Ah internal resistance measurements are very similar for the group that were cryogenically frozen (DK01 – blue trace) and the control group (DK02 – red trace). Table 4 summarises the Panasonic 3 Ah internal resistance *t*-test results, which are all > 0.05, therefore we accept the null hypothesis and conclude that both groups have the same means. Flash cryogenic freezing does not affect the internal resistance of both DK 5 Ah and Panasonic 3 Ah throughout the automotive useful life.

Table 3
DK 5 Ah capacity *t*-test results.

Cycles	*t*-test	Cycles	*t*-test	Cycles	*t*-test	Cycles	*t*-test	Cycles	*t*-test
0	0.19	469	1.00	1309	0.34	1834	0.69	2359	0.26
75	0.33	574	0.84	1414	0.38	1939	0.71	2464	0.20
150	0.57	679	0.94	1519	0.36	2044	0.67	2569	0.24
259	0.58	1099	0.45	1624	0.38	2149	0.40	2464	0.27
364	0.78	1204	0.40	1729	0.38	2254	0.38		

Table 4
Panasonic 3 Ah capacity *t*-test results.

Cycles	*t*-test	Cycles	*t*-test	Cycles	*t*-test	Cycles	*t*-test	Cycles	*t*-test
0	0.64	180	0.54	360	0.77	630	0.75	810	0.62
45	1.00	225	0.64	405	0.58	675	0.84	855	0.61
90	0.28	270	0.83	540	0.57	720	0.63	945	0.28
135	0.12	315	0.86	585	0.64	765	0.22		

3.2.1. Internal resistance measurements after cycling

The six plots in Fig. 3 show an apparent reduction in internal resistance during the cycling at the fifth measurement. This corresponds with the pause in testing (see Section 3.1.1) and is exacerbated by the HPPC measurementsnot all being performed at the desired SOCs (80,

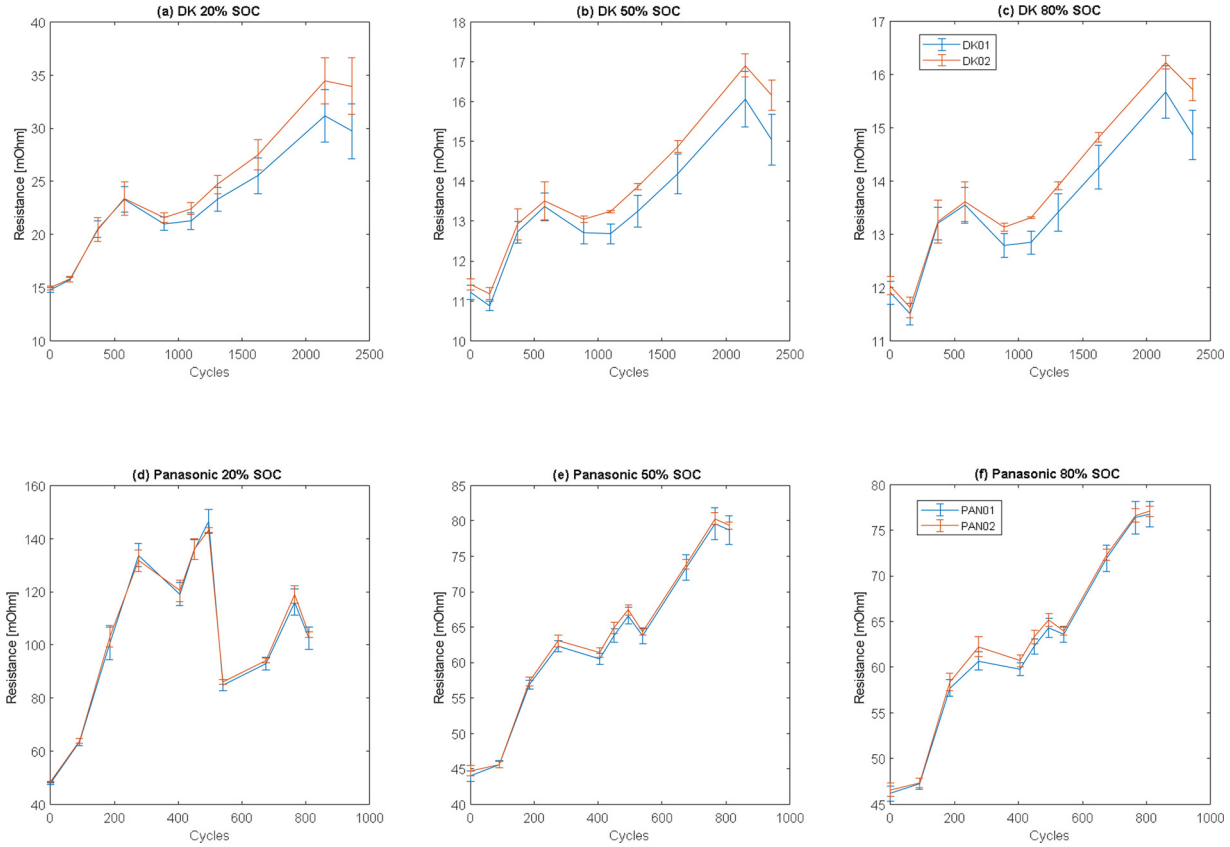

Fig. 3. HPPC measurement of DK 5 Ah cells at (a) 20% SOC, (b) 50% SOC, and (c) 80% SOC, and Pan 3 Ah cells at (d) 20% SOC, (e) 50% SOC, and (f) 80% SOC, after cycling (control group in red). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Table 5
Panasonic 3 Ah and DK 5 Ah estimated SOC for HPPC measurements.

Cycles	Panasonic 3 Ah estimated SOC			Cycles	DK 5 Ah estimated SOC		
0	79.9%	49.7%	19.6%	0	82.1%	55.3%	28.6%
90	79.2%	47.9%	16.7%	150	81.8%	54.5%	27.2%
185	77.7%	44.3%	10.8%	370	81.2%	53.0%	24.8%
275	77.3%	43.3%	9.3%	580	80.9%	52.4%	23.8%
405	76.7%	41.9%	7.0%	889	80.4%	51.1%	21.8%
450	76.8%	42.1%	7.3%	1099	81.8%	54.6%	27.3%
495	76.8%	42.1%	7.3%	1309	81.4%	53.6%	25.7%
540	80.0%	50.0%	19.9%	1624	81.8%	54.5%	27.3%
675	80.0%	50.1%	20.1%	2149	81.9%	54.8%	27.8%
765	77.8%	44.6%	11.4%	2359	81.8%	54.5%	27.2%
810	80.2%	50.4%	20.7%				

50, and 20%). Table 5 shows the estimated SOC that the HPPC measurements were carried out for both the cryogenically frozen cells and the control group. The SOCs are estimated since the capacity measurements used to establish the remaining capacity are affected by reversible effects (see Section 3.1.1 for further details).

Since the cryogenically frozen cells and the control group were both measured at the same SOCs, the authors assert that the SOC variation does not detract from the findings reported, that is to say the flash cryogenic freezing does not affect the internal resistance of DK 5Ah and Panasonic 3Ah cells.

3.3. Further work

No detrimental effect to cell performance was found on the flash frozen cells for two cell chemistries and form factors (18,650 energy NCA and small pouch power NMC). It is expected this applies to other Li-ion chemistries, such as lithium Manganese Oxide ($LiMn_2O_4$) and lithium iron phosphate ($LiFePO_4$). However, the transferability of these results to other LIB technologies requires further experimentation before this can be more fully understood. Cell autopsies where the electrodes are removed from the cell in order for the electrode surface to be analysed with a scanning electron microscope are to be performed in order to confirm the results.

Further work is being undertaken to scale the work from cell to module and pack level. Since most state-of-the-art electrolytes crystallise at temperatures below −40 °C [27], it is expected that it is not necessary to maintain LIB packs at cryogenic temperatures (below −150 °C) in order to prevent thermal runaway. As such, further experiments are being undertaken to establish the minimum temperature to prevent thermal runaway within a complete battery installation to facilitate safe transportation. Different freezing rates will be investigated in order to establish how it affects the results. Finally, once proof-of-concept has been established, the authors recommend a full economic assessment of the methodology that will further underpin commercialisation and adoption by industry.

4. Conclusions

The experiment evidences presented implies that flash freezing Li-ion cells causes no significant detrimental effects on cell electrical performance throughout the whole automotive life (20% capacity fade). The cell performance was determined by its impedance and energy capacity throughout cycling, as these dictate the power delivery capability and the amount of energy that can be stored, which in turn, defines the EV range, acceleration, and charging performance. Cell impedance and capacity were measured at regular intervals during cycling to quantify any effects of flash freezing on cell ageing and performance degradation. No statistical difference, with a 95% confidence level, in cell performance was found on two cell chemistries and form factors (18,650 energy NCA and small pouch power NMC) between the cells that were flash frozen and the control groups. This provides initial confidence that flash cryogenic freezing will not affect battery lifetime and ageing.

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