

1 Case Report

2 Looking Deeper into the Galaxy (Note 7)

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12 **Abstract:** Li-ion cell designs, component integrity and manufacturing processes all have critical
13 influence on the safety of Li-ion batteries. Any internal defective features that induce a short circuit,
14 can trigger a thermal runaway: a cascade of reactions, leading to a device fire. As consumer device
15 manufacturers push aggressively for increased battery energy, instances of field failure are
16 increasingly reported. Notably Samsung made a press release in 2017 following a total product
17 recall of their Galaxy Note 7 mobile phone, confirming speculation that the events were attributable
18 to the battery and its mode of manufacture. Recent incidences of battery swelling on the new iPhone
19 8 have been reported in the media, and the techniques and lessons reported herein may have future
20 relevance. Here we look deeper into the key components of one of these cells and confirm evidence
21 of cracking of electrode material in tightly folded areas, combined with a delamination of surface
22 coating on the separator, which itself is an unusually thin monolayer. We report microstructural
23 information about the electrodes, battery welding attributes and thermal mapping of the battery
24 whilst operational. The findings present a deeper insight into the battery's component
25 microstructures than previously disseminated. This points to the most probable combination of
26 events and highlights the impact of design features, whilst providing structural considerations most
27 likely to have led to the reported incidences relating to this phone.
28

29 1. Introduction

30 Lithium-ion cells (LIBs) are statistically safe, with a 1 in 10 million failure rate established during
31 the manufacturing (rather than due to field ageing) and such failures are considered an exception
32 and not a reliability problem [1]. However, with billions of cells in use globally failures are to be
33 expected and are a cause of health and safety concern. To eliminate these effectively, we need
34 techniques to fully understand their root causes [2].

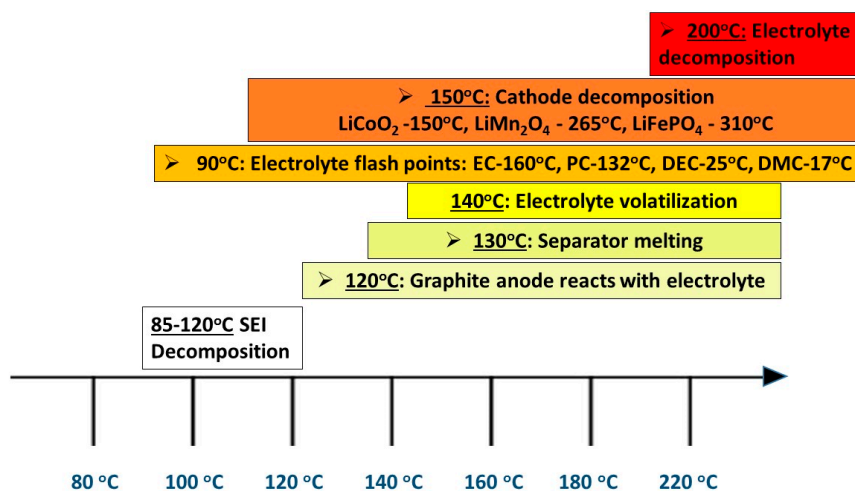
35 In 2006, a one-in-200,000 failure rate triggered a recall of almost six million lithium-ion battery
36 packs used in several brands of notebook computers [3]. Sony, the maker of the lithium-ion cells in
37 question, pointed out that on rare occasions microscopic metal particles may come into contact with
38 other parts of the battery cell, leading to a short circuit within the cell [1]. In 2013 Boeing had its
39 newest and most advanced aircraft, the 787 Dreamliner, globally grounded due to incidences of fire
40 that emanated from a battery powering an APU (auxiliary power unit) on board flight JA829 [4–6].
41 At least 3 other reported incidences have affected other airliners with a significant fleet of 787 aircrafts
42 [7]. There is a continued drive for faster innovations in Li-ion cell formats, regardless of type and
43 dimension, to meet advanced performance criteria. We propose that effective component
44 characterisation is critical over multiple scales if we are to meaningfully interpret and reduce failure
45 rates.
46

47 Within Li-ion cells an internal short circuit can generate sufficient heat to initiate exothermic side
 48 reactions involving energy dense and sometimes volatile components within the cell – when these
 49 proceed out of control they cause a thermal runaway resulting in fires [8]. Additional electrical,
 50 mechanical or thermal abuse conditions may also contribute to such a reaction cascade in certain circumstances
 51 and be associated with thermal runaway to adversely affect Li-ion cells [9]

52 1.2 The thermal runaway cascade.

53 The primary functional components of a lithium-ion cell are the anode and cathode (each taking
 54 the form of a metallic foil coated with electrochemically active components) and electrolyte (typically
 55 a liquid organic compound). During the charge process Li ions move from the cathode through the
 56 separator to the anode, whilst electrons flow through an external circuit. Causes of short circuiting
 57 may be external or internal. The latter may include foreign contaminants or Li-dendrites penetrating
 58 through the separator (an insulating porous film separating anode and cathode). Anything that
 59 results in electrical contact between anode and cathode [10] will result in a short circuit.

60 Prior to commercial availability a battery will undergo a formation cycle. This allows for the
 61 creation of a protective layer on the electrodes, generated from the decomposition of a small amount
 62 of electrolyte solution on the surface of the electrodes. This is termed the *solid electrolyte interphase*
 63 (SEI). Following a short circuit event the localized temperature inside the cell begins to rise and at
 64 100 °C the SEI film begins to decompose [11]. A series of exothermic reactions follow between SEI
 65 compounds and electrolyte solvents such as ethylene carbonate (EC) and diethyl carbonate (DEC).
 66 When this cascade of reactions proceeds uninterrupted it causes a rapid elevation of the internal
 67 temperature whereby a series of reactions can generate large volumes of flammable hydrocarbon
 68 gases, which will vent to atmosphere - see Figure 1 [12–15].



69 **Figure 1.** A summary of the thermal runaway cascade of reactions that take place inside a lithium-ion cell as a function of temperature

70

71 Cathode oxide materials can reduce in exothermic disproportionation reactions e.g. lithium
 72 cobalt oxide breakdown outlined in Equation 1 [13,16]. Charged Li_xCoO_2 (lithiated) can decompose,
 73 releasing oxygen at elevated temperatures¹⁷. Equation 1 shows the complete reduction to cobalt metal
 that will only occur if sufficient reducing agent (solvent) is present [16].

74



75

76 Therefore in the context of the continued widespread use of this cathode material, it will
 77 potentially be problematic only when conditions warrant thermal stability, as compares with e.g.
 78 lithium iron phosphate [18]. The choice of electrode chemistries is often a question of energy density
 vs. rate requirements and such things can often result in trade-offs that dictate ultimate performance.

79

80 Investigating and understanding the root causes of cell failures allow us to re-engineer the way
81 cells are constructed or manufactured to then mitigate the safety hazards [19] associated with the
82 likelihood of internal short circuits in LIBs. It is not straightforward to capture all failure origins in
83 batteries as they are closed systems. *In operando* characterisation techniques have become a popular
84 area in battery research and are proving to be very valuable. They allow us to measure or visualise
85 occurrences in real-time, without stopping operation and opening the battery. This is a very high
86 priority and active area within the energy storage research community [20].
87

88 **1.3 How was battery manufacturing the cause of the faults?**

89
90 In 2016 there was a total product recall of the Samsung Galaxy Note 7 which has cost the
91 company an estimated \$17 billion [21], with the reported cause of the fire incidences claimed to be
92 attributable to a “non-optimized manufacturing process relating to the battery.” The official
93 investigation by Samsung highlighted several key areas to which the phone fires were attributed. The
94 errors reported were both in the design and manufacturing of the batteries and were reported to
95 include:
96

97 *(i) Insufficient insulation material within the batteries; (ii) Not enough room to safely accommodate the battery;*
98 *(iii) Welding burrs on the positive electrode resulting in penetration of the insulating tape (on the tabs) and*
99 *separator.*

100

101 Additional independent investigations by 3 organisations; UL, Exponent and TÜV- Rheinland
102 analysed hundreds of cells and added more insight into root cause analysis and abuse approaches
103 [22]. Their respective press releases expanded on the suspected failure modes reported from the 2
104 battery companies Manufacturers A and B, used to supply the cells for the Note 7 manufacture.

105 **1.4 Reported Failure Suspicions & Battery Component Causes.**

106 Two additional press releases presented investigations of batteries from 2 different
107 manufacturers [23,22]. Cells from Manufacturer A and B were described in the Root Cause Analyses
108 reports, citing that the thermal failure events were due to unintended anode deformation in the
109 corner of the cell closest to the negative tab. Cells from Manufacturer B were reported to have no such
110 deficiencies but were reported to contain welding defects tall enough to bridge the distance to the
111 anode tab. Additionally insulating tape was found to be missing over the cathode electrode tab.

112 The second investigation tested 10 devices containing cells from Manufacturer A alongside 10
113 from Manufacturer B and summarized the suspected causes of the thermal failures as a combination
114 of:
115

116 *(i) Internal short circuit (ISC) at the upper right corner of the cells; (ii) Repeated deformation of the separator*
117 *at corner locations; (iii) Missing insulating tape on the cathode tab; (iv) Poor alignment of components; (v)*
118 *Uneven charge status and (vi) Thinner separator compared with others used in previous devices.*

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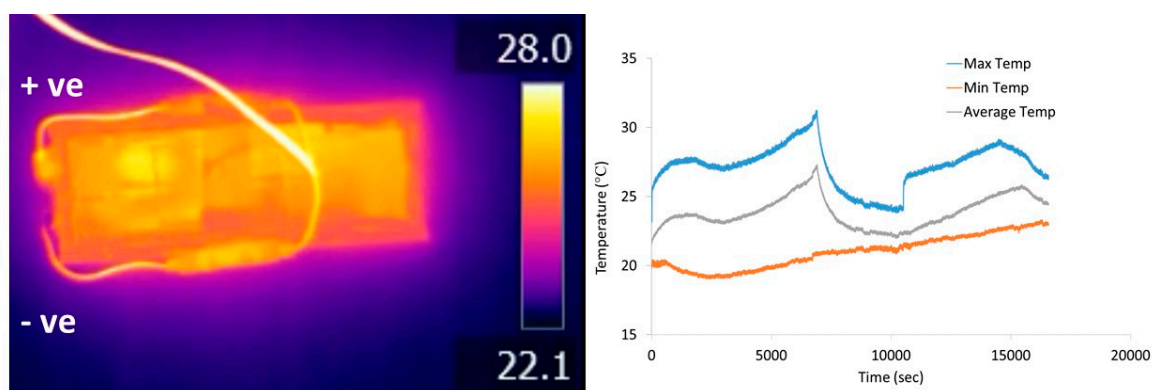
120 This study seeks to corroborate the reported findings by elucidating - across multiple scales – all
121 the microstructural aspects inside the battery but to add to the component and material-level details
122 and resolution. We capture deeper analysis of key components compared with the press releases
123 issued. Several dimensional levels will be considered.
124
125

126 **T2. Results & Discussion**

127 **2.1 Electrochemical Characterisation.**

128 Figure 2(a.) shows the temperature profile (maxima and minima) as a function of charging time
 129 with periodic thermal imaging. For the duration of the charging step the temperature of the cell did
 130 not rise above 32 °C at a charging rate of C/2.

131 A noticeable temperature difference of up to 3 °C can be seen across the cell from top to bottom.
 132 The higher temperature side is where the positive terminal is sited and this is where current transport
 133 occurs with the cathodes. The current collectors for this electrode are made of aluminium, whereas
 134 the anode current collectors are made of copper. Cu and Al have differing thermal conductivities of
 135 401 and 205 W/ (m K) and electrical resistivities of 1.72×10^{-8} and $2.65 \times 10^{-8} \Omega\text{-m}$ respectively, hence
 136 the anode has lower electrical resistance and the capacity to dissipate heat more quickly. Additionally
 137 the Al current collector is 5 μm thicker than the Cu current collector at the anode and can thus
 138 contribute to slowing down heat transfer.
 139



140 **Figure 2** – Operational temperature profile with thermal imaging at 180 minutes charging.

141 2.2 X-ray CT Characterisation of Device & Internal Battery Features.

142 X-ray micro-tomography scans were performed at 3 levels: (a) the intact mobile phone, (b) the
 143 battery and (c) the battery components, which are the electrodes and separator. Figure 3 (a.) shows
 144 X-ray tomographic scans of regions of the cell within the device.

145 (b) Battery Cell Characterisation

146 Consistent with device findings ascribed to cells that were produced at Manufacturer A, from
 147 the enlarged images in Fig 3 a. the feature that resemble a fallen row of dominoes the bottom section
 148 edge of the prismatic from unintended damage to the negative electrode windings. The corners are
 149 folded over and thought to be caused by a pouch design with insufficient internal space around
 150 certain regions of cell content. There is a clear deformation at the cell here, thought to derive from the
 151 manufacturing and assembly process.

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 153 The potential implications of this, taking into account typical expected dimensional
 154 manufacturing tolerances, are to increase the likelihood of an internal cell short circuit and
 155 subsequent thermal failure during normal operation.
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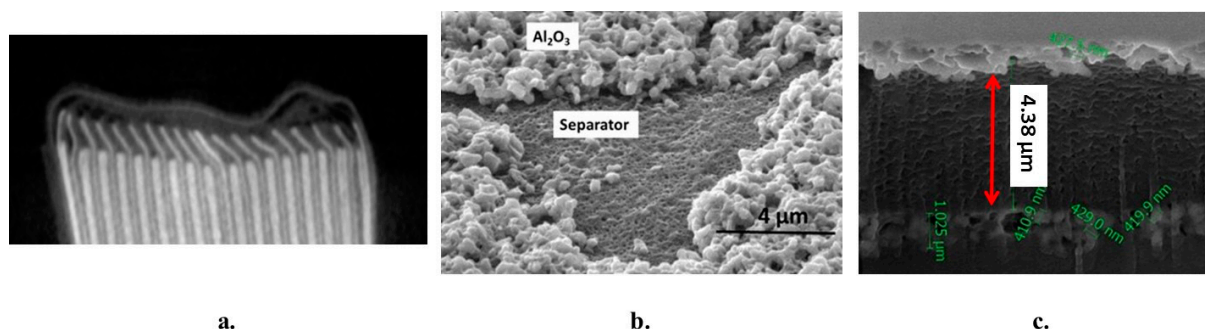


Figure 3 (a.) X-ray CT scan of edge deformations in the phone battery electrodes; (b.) SEM image of ceramic-coated separator revealing a delaminated region (c.) Cross-sectional SEM image of the separator.

157

158 (c) Cell Component Characterisation

159 Upon forensic disassembly of the cell and subsequent unravelling of the electrodes there were
 160 visible areas of coating irregularities on both the electrodes and the separator – see Figure 3 b. On the
 161 anode there is coating delamination on several edge regions, with cracking of the coating at the folded
 162 areas. This reflects greater pressure exerted on these regions and could indicate the potential for
 163 separator damage in these zones, leading to internal short circuit. Additionally, any material
 164 detached from the electrode, if it were to enter the electrode stack, could increase local mechanical
 165 pressure on the separator.

166 Conversely, on the cathode electrode there were frequent deposits of a ceramic layer – this is
 167 investigated further. There have been no mentions of electrode manufacturing quality concerns or
 168 separator coating issues in the previous reports. SEM imaging was carried out on electrodes and
 169 pieces of separator material and is depicted in Figure 4, which shows surface analysis and cross-
 170 sectional characterisation. The anode was mainly composed of graphite and the cathode was based
 171 on lithium cobalt oxide (LiCoO_2).

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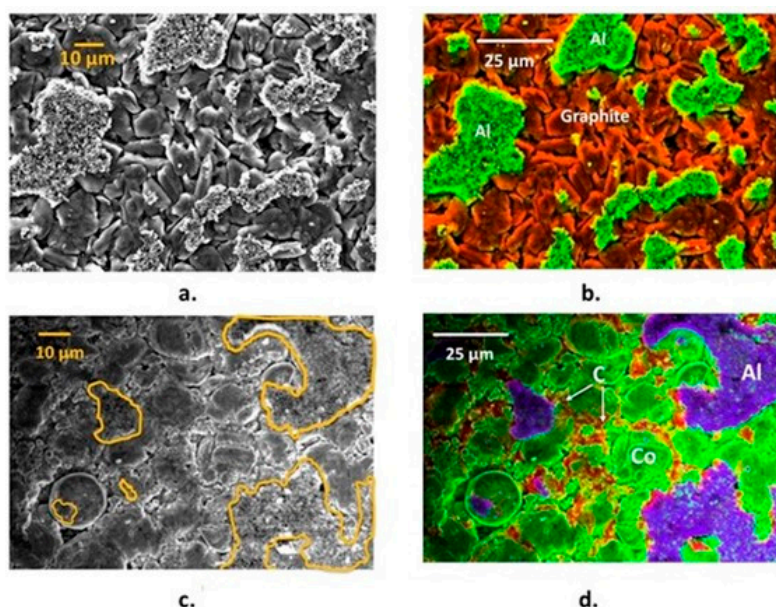


Figure 4 – SEM-EDS images of (a.) LCO cathode (b.) Graphite anode (c.) Cathode showing carbon nanotubes and (d.) Cathode broad particle size distribution.

173

174 The separator is a critical component in a battery cell and is a porous polymer membrane used
 175 to create an electrically non-conducting physical barrier between the anode and cathode.

176 Mechanical failure of this component can result in catastrophic failure of the cell and often occurs
 177 from puncture or thermal shrinkage of the membrane [24]. Separators have a complex anisotropic
 178 structure and can be susceptible to softening effects by the organic electrolyte solvents [25].

179 Such interactions can be characterized by calculation of the Flory-Huggins interaction
180 parameters when the polymer identity and electrolyte solvent composition is known. Commonly
181 used separators are based on multiple layers to provide mechanical resilience and a thermal
182 shutdown mechanism for additional safety. The polymer portion of the Samsung separator is a
183 ceramic coated monolayer and was less than 5 μm in thickness with a double-sided covering of Al_2O_3
184 of around 1 μm thickness. Other commercially available separators such as Celgard 2325 trilayer
185 polyolefin separators (polypropylene – polyethylene -polypropylene), are over four times the
186 thickness of this at 25 μm , and quote product thickness ranges of 12 – 40 μm [26]. Multi-layer
187 polyolefins can be manufactured with a shutdown function and used as a fail-safe component in
188 batteries. However, reports of extremely low wettability with liquid electrolytes have limited its
189 further application in energy storage systems for electric vehicles [27,28]. Ceramic coatings have
190 successfully demonstrated resistance to thermal shrinkage in batteries in operating temperature
191 ranges of 110 – 200 $^\circ\text{C}$ for 30 min [29].

192 On disassembly of the cell and subsequent unravelling of the components there were areas of
193 separator that had small sections of electrode coating adhered to them. A thin Al_2O_3 coating is evident
194 on the polymer separator and had become delaminated in parts – as can be supported by the ceramic
195 fragments found on the electrode surfaces in Figure 4. This was apparent during the disassembly of
196 the pouch cell with electrode patches adherent to some areas near the edges of the separator,
197 suggesting more mechanical stress exposure in these areas. From the SEM images below, the ceramic
198 material phases are identified as being aluminium oxide from the adjacent EDS chemical maps. The
199 electrode active materials were found to be graphite and lithium cobalt oxide for the anode and
200 cathode respectively (as expected). The particle size range is very broad in the cathode allowing for
201 increased packing density of the active material and thus higher capacity.

202 *Characterisation of Welded Joints in Battery Tabs*

203 Figure 5 shows SEM images of the anode and cathode welded tab regions that transport current
204 (charge) to the battery electrodes. This is typically used because conventional fusion welding
205 processes (such as resistance spot welding and laser welding) are faced with challenges in joining
206 multiple sheets of highly conductive, dissimilar materials over large areas [30]. The fusing together
207 of cell current collectors and tabs achieved by ultrasonic welding uses high-frequency energy ≥ 20
208 kHz to generate oscillating shears at the interface between a sonotrode and metal sheets. This
209 produces solid-state bonds between the sheets clamped under pressure in a period of time less than
210 a second [31]. Thus, the solid-state mechanism of the ultrasonic process is better suited to generating
211 weld joints with higher integrity.

212 Evident within this type of welding is material flow whereby excessive plastic deformation can
213 thin and negatively affect the weld quality [30]. This can result in pieces of material from the thermos-
214 mechanically affected zone (TMAZ), becoming detached and the weld bond being discontinuous.
215 Cells from Manufacturer B were reported to have sharp protrusions in the welded tab region of the
216 cathode, in the region of the thermomechanical affected zone – see Figure 6 for a description of key
217 attributes in ultrasonic welds. UL and Exponent released independent reports of their testing on the
218 Note 7 and reported internal cell faulting between the positive electrode tab welding defects and the
219 anode copper foil directly opposite the defective welds – depicted in figure 7 [32]. The figure shows
220 sharp and relatively tall welding defect features and the report suggested a short circuit occurred
221 between the weld defect features on the positive tab and the copper of the negative electrode,
222 resulting in thermal runaway at high states of charge [32].

223 The weld areas in the cathode tab section characterised in this study did not appear as sharp
224 protrusions, however our sample size was limited and such failure, if present, would be expected
225 only on a statistically small proportion of cells. We did however detect areas of unbonded weld
226 regions and one of the assessment criteria of weld quality is correlated to the joint characteristics. An
227 ultrasonic metal weld with good weld quality should have dense and continuous interfacial bonds
228 [30].

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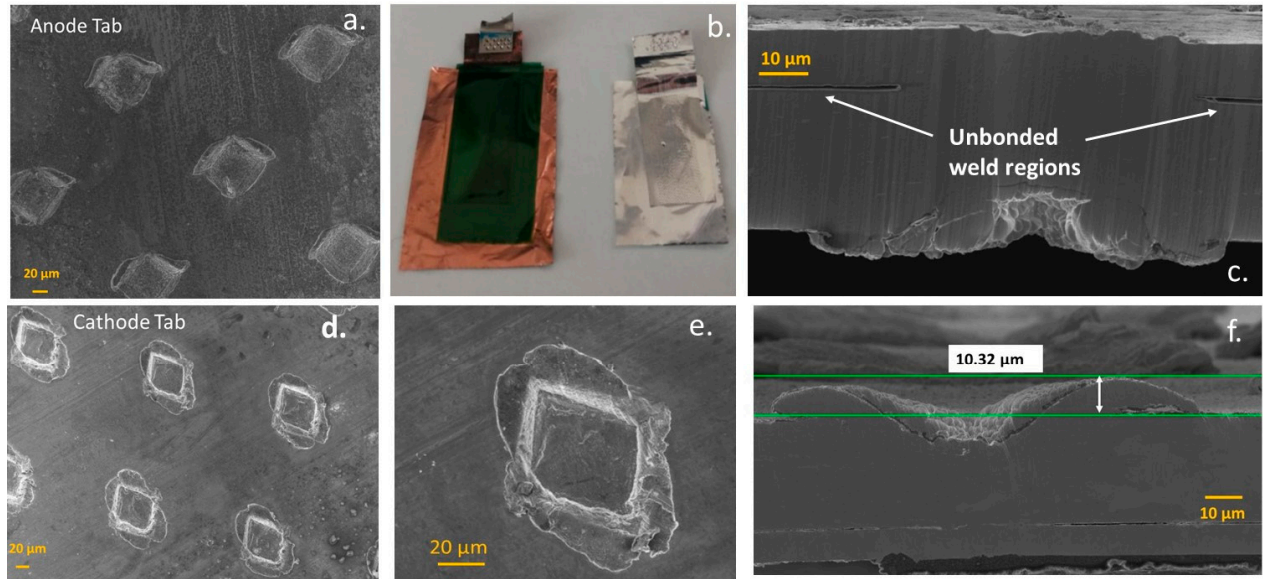


Figure 5 – Images showing (a.) Lower magnification SEM of welded tab (b.) Missing insulating polyimide tape on the cathode tabs, (c.) SEM cross-section of an anode tab weld (d.) Lower magnification SEM of the cathode welded tab (e.) Higher magnification cathode weld (f.) SEM cross-section Ion-milled cross-section of a cathode tab weld.

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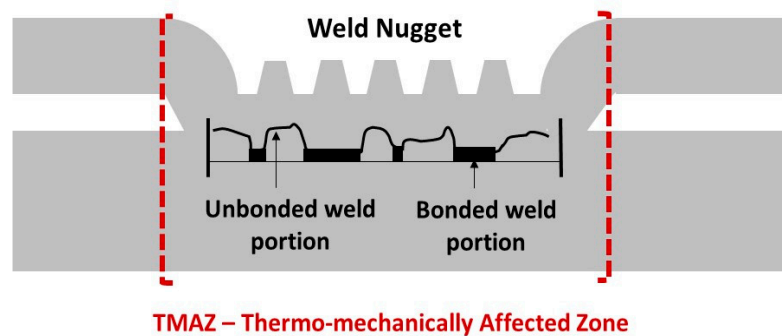


Figure 6 – Schematic of some of the structural attributes of an ultrasonic weld

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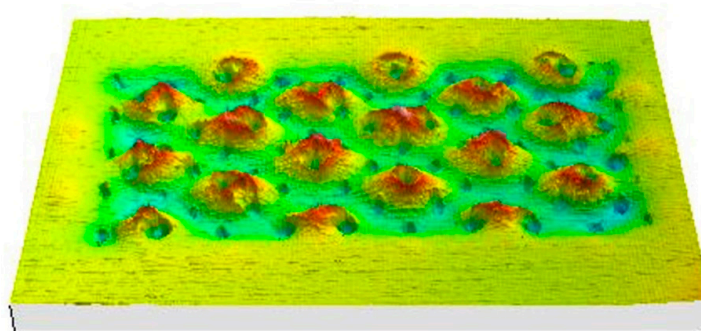


Figure 7 – 3D reconstructed X-ray tomographic scan weld area defect features in the positive tab from Manufacturer B cells.

237

238 The quality and reliability of Li-ion battery tabs is of significant importance especially as batteries are
239 becoming increasingly energetic. A battery tab joint can cause the failure of an entire battery pack if
240 it yields during operation [33]

241 **3. Materials and Methods**

242

243 **3.1 Electrochemical Characterisation.**

244 The charge-discharge parameters were firstly characterised on the phone in the as received
245 condition. The cell voltage was around 1.5 V and the cell was rated as 4.4 V maximum. The initial
246 electrochemical characterisation test used a 0.4 amp charge current, applied until the cell reached 4.0
247 V. No unusual cell behaviour was observed during this first charge.

248 **3.2 Charging limitation from “safety software patch”**

249 Our investigations were conducted using a Samsung Galaxy Note 7 phone, bought in the open
250 market shortly after the announced product recall. Samsung launched an over-the-air safety software
251 patch in Sept 2016 which limited the extent to which the phone could be charged, and encouraged
252 owners to submit their devices for recall This was set to 2450 mAh; 70 % of its 3500 mAh capacity,
253 and reduced further to 30 % capacity (1050 mAh) in December 2016. To prevent this software from
254 affecting our experiments, the phone was housed in a makeshift Faraday cage during initial
255 operation.

256

257 **3.3 X-ray Computed Tomography.**

258

259 The method firstly analysed the Region of Interest (ROI) inside the whole sample without
260 dismantling the device, scanning both the uncharged and partially charged battery. The tomography
261 scanners used for this analysis were: X-Tek X-H 225/320 LC (Nikon Metrology, UK) using CT
262 reconstruction software CT Pro 4.3 (Nikon Metrology, UK) and inspection software VG Studio Max
263 2.2 (Volume Graphic GmbH). The phone was scanned twice: (i) with the battery discharged, and (ii)
264 with the battery partially charged. The device was positioned consistently to maximize magnification
265 and reduce potential artefacts.

266

267 In order to obtain a satisfactory resolution, the device was scanned in 2 phases –“Top and Bottom”
268 and stitched together using a VG Studio Max Best Fit algorithm. Conditions for the top and bottom
269 scans were the same with the individual scans of the battery partially charged having slightly
270 different settings to provide optimum results. A fixture was used in all of the scans to ensure the
271 position of the device and avoid micro movement during the 360 degrees rotation. The series of
272 variables selected are based on the scanned material, the capabilities of the CT system and
273 the required resolution. The settings and the positioning of the part was selected for maximum
274 magnification resulting in a voxel size and resolution of 52µm. Materials with noticeably
275 different atomic masses (density) create beam hardening and in order to reduce the error a
276 hardening reduction algorithm was applied

277 **4. Conclusion**

278 The primary purpose of our activity was to develop and combine a suite of forensic investigation
279 techniques to detect and characterise failure modes in LIBs. We analysed key components within the
280 battery from the Galaxy Note 7, as a topical example, to validate the company’s and the press release
281 findings, but also to gain deeper structural insight into the most likely routes to the incidences. From

282 the tomography scans around the edges of the battery it appears that the cell which we analysed was
283 derived from Manufacturer A: Samsung SDI. Thermal measurements of the cell under charge and
284 discharge showed temperature differentials within the cell which are within expected ranges, and
285 gave no evidence that thermal design was a contributory factor. Our findings corroborate several of
286 the features reported to be the cause for recall, but in addition, use of forensic disassembly and
287 microscopy reveals electrode damage in tightly folded areas, and a weak interfacial bonding of the
288 ceramic layer on the separator as being potential contributory factors.

289 X-ray tomography confirmed reported findings of the upper corner deformation and
290 compression of the battery contents, leaving minimal volume within the pouch using this type of cell
291 design. Forensic disassembly revealed electrode coating damage in edge and fold regions that were
292 subjected to almost 360° bending. Such localised defects can induce local lithium deposition and
293 dendrite growth, and the plating of lithium could trigger a short circuit through penetration of a thin
294 separator (with lower levels of tensile resilience compared with other commercially used thicker
295 films). This is especially the case in areas where delamination of the ceramic coating had occurred,
296 which were quite frequent over the areas examined. That the ceramic coating became detached
297 during disassembly (following minimal cycling) suggests a weak bonding to the polymer and low
298 interfacial integrity. That the ceramic adheres in some places to the electrode, and in others to the
299 separator, may be significant. For instance, if these layers were to experience micro-slip relative to
300 each other, as the cell expands and contracts due to electrical and thermal cycling, this could result
301 in areas of separator devoid of ceramic coating. Additionally there would be a build-up of the ceramic
302 coating thickness on others, increasing mechanical pressure on the separator. This may manifest itself
303 during assembly or in use.

304 Overall, it is clear that the industrial push for increasing energy density of batteries to allow
305 more powerful phones to operate for acceptable periods between charging is resulting in engineering
306 compromises to cell design and component specification which have the potential to deliver such
307 advances at the expense of safety. Industrial actors such as Samsung take this responsibility
308 extremely seriously and devise test and development methods to control such risks to acceptable
309 levels. The body of this work demonstrates the value of combining X-ray tomography, forensic
310 disassembly and microscopy to elucidate failure pathways in support of such test and development
311 methods. Further work by the authors is planned to investigate further combination with techniques
312 addressing localised electrochemical characterisation and thermo-mechanical loading.

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