CELL-INTEGRATED SENSING FUNCTIONALITIES FOR SMART BATTERY SYSTEMS WITH IMPROVED PERFORMANCE AND SAFETY

GA 957273

D5.3–RECYCLING ASSESSMENT AND INTEGRATED VALIDATION

LC-BAT-13-2020 - Sensing functionalities for smart battery cell chemistries

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Summary

The deliverable D5.3. reports the recyclability of lithium-ion battery (LIB) cells with built-in SENSIBAT Level-1 and Level-2 sensors. It summarizes the activities related to Task 5.3 in the frame of work package 5.

In general, the recycling process of LIBs is significantly influenced by the energy stored in these cells, as handling, storage and the preparation for recycling must show considerations for potential environmental and safety related harms.

Next to security risks, the example of an industrialized recycling process, further provides evidence why industrial disassembly processes are not feasible for small-scale LIBs and related modules. Regardless the disassembly process, the deliverable identifies the recycling routines that are applicable for each main component of LIBs.

Based on results obtained in the testing (T5.1) and from the cost benefit assessment (T5.2), the deliverable includes an overall technical, economic, manufacturing and (limited) environmental assessment for Level-1 and Level-2 sensors. This assessment also includes considerations by the advisory board and outcomes of the report on Key Exploitable Results (KER). In this context, the document also provides information on the foreseeable future application fields for the developed sensors.

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Abbreviations

Symbol / Abbreviation	
2LB	*Second-life battery*
CO	*Carbon monoxide*
C_6H_6	*Benzene*
C_8H_8	*Styrenics*
CMC	*Carboxymethyl cellulose*
DEC	*Diethyl Carbonate*
DMC	*Dimethyl Carbonate*
EC	*Ethylene Carbonate*
EMC	*Ethyl Methyl Carbonate*
HCl	*Hydrochloric acid*
HF	*Hydrofluoric acid*
LIB	*Lithium Ion Battery*
$LiPF_6$	*Lithium hexafluorophosphate*
LCO	*Lithium Cobalt Oxide, $LiCoO_2$*
LFP	*Lithium Iron Phosphate, $LiFePO_4$*
LMO	*Lithium Manganese Oxide, $LiMn_2O_4$*
LTO	*Lithium titanate*
NCA	*Lithium Nickel Cobalt Aluminum oxide, $LiNi_xCo_yAlzO_2$*
NiCd	*Nickel Cadmium*
NiMH	*Nickel Metal Hydride*
NMC	*Lithium Nickel Manganese Cobalt Oxide, $LiNi_xMn_yCozO_2$*
PC	*Propylene Carbonate*
PE	*Polyethylene*
PI	*Polyimide*
PP	*Polypropylene*
PTC	*Positive Thermal Coefficient*
PVdF	*Polyvinylidene difluoride*
SoH	*State of Health*
SoS	*State of Safety*
VOC	*Volatile Organic Compounds*

1 Introduction

The present deliverable 5.3 contains the recycling assessment of cells used within the SENSIBAT project. This comprises also the materials and elements of the incorporated sensors. The deliverable concludes with a technical, economic, manufacturing and environmental assessment.

2 Recycling Assessment

2.1 Recycling Assessment of 1Ah and 5Ah baseline cells

2.1.1 General Overview and material characteristics

Within the emerging usage of lithium-ion batteries (LIBs), the number of spent LIBs increases and will increase further in the following years. Accordingly, alongside second-life applications of "aged" LIBs, close attention must be paid to the recovery of valuable material resources by recycling these cells. Precious resources include EU's critical and strategic raw materials, *e.g.*, lithium, nickel and cobalt.

Unfortunately, materials of LIB are inherently hazardous (e.g., flammable components, toxic decomposition products). This comes with serious challenges in waste management due to potential safety risks arising from abusive disposal and storage.

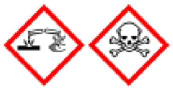
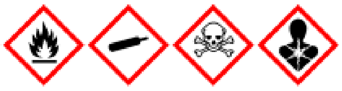
Examples of cell components and their mass fractions in LIBs are listed in Table 1:

Table 1: Exemplary commercial LIB cell composition (SENSIBAT components underlined) (1)

Battery component	**Materials**	**Mass-%**
Housing	Steel or Aluminium	20-25
Cathode (positive electrode)	LCO, NMC, NCA, LFP or LMO	25-35
Anode (negative electrode)	Graphite	14-19
Electrolyte	LiPF6 dissolved in PC, EC, DMC or DEC	10-15
Cathode current collector foil	Aluminium	5-7
Anode current collector foil	Copper	5-9
Separator	PP, PE	1-4
Others (additives)	Carbon black, silicon, etc.	Balance

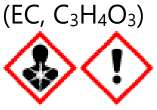
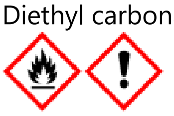
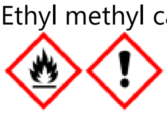
Clearly, LIBs have a more complex component build-up than previous battery systems (lead acid, NiCd, NiMH, alkaline, *etc*.). For some of these battery systems, *e.g.*, water-based electrolytes and relatively simple electrochemical reactions at the electrodes result in less severe effects caused by thermal or mechanical abuses. Instead, LIBs contain volatile, flammable organic electrolytes and fine solid particles (graphite and metal oxide), whose reactivity (associated to gas evolution), leakage or exposure is associated with the risks of fire and pollution. Table 2 report the toxicological and environmental risks of gases released from LIBs in presence of undesired decomposition of electrode materials and electrolytes.

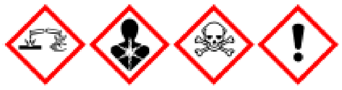
Table 2: Environmental and human health risks of gases released from LIBs (2)

Reaction product Properties according to GHS	Reaction product Properties according to GHS
Benzene (C_6H_6) 	H225 H304 H315 H319 H340 H350 H372 H412 Single lethal dose of benzene for humans: 125 mg/kg (= 10 ml/70 kg)
Styrenics (C_8H8) 	H226, H315, H319, H332, H372, H361d Ingestion: LC50, for rats: 5000 mg/kg, in mice: 316 mg/kg
Hydrofluoric acid (HF) 	H300, H310, H330, H314, H318 lethal dose: 20 mg/kg body weight.
Hydrochloric acid (HCl) 	H300, H310, H330, H314, H318 30-min LC50, in rats: 4700 ppm, in mice: 2600 ppm
Carbon monoxide (CO) 	H220, H331, H372, H360d 30-min LC50, in humans: 3000 ppm

The environmental risks inherently associated with the LIB materials are instead reported in Table 3.

Table 3: Environmental and human health risks of materials used in commercial LIBs (3)

Material	Properties
Ethylene carbonate (EC, $C_3H_4O_3$) 	Vapor pressure: 21 Pa (20 °C). Toxicity and hazard to water: • Causes serious eye irritation • slightly hazardous to water (German water hazard class: WGK 1*)
Propylene carbonate (PC, $C_4H_6O_3$) 	Vapor pressure: 4 Pa (20 °C), 130 Pa (50 °C). Toxicity and hazard to water: • Causes serious eye irritation • slightly hazardous to water (WGK 1)
Dimethyl carbonate (DMC, $C_3H_6O_3$) 	highly flammable, highly volatile Vapor pressure: 5300 Pa (20 °C). Toxicity and hazard to water: • irritant effects, nausea, intoxication, unconscious-ness, respiratory stop • slightly hazardous to water (WGK 1)
Diethyl carbonate (DEC, $C_5H_{10}O_3$) 	highly flammable, highly volatile Vapor pressure: 1100 Pa (20 °C). Toxicity and hazard to water: • low toxicity • slightly hazardous to water (WGK 1)
Ethyl methyl carbonate (EMC, $C_4H_8O_3$) 	highly flammable, highly volatile Vapor pressure: 3600 Pa (25 °C). Toxicity and hazard to water: • irritant

	• slightly hazardous to water (WGK 1)
Lithium hexafluorophosphate ($LiPF_6$) 	Toxicity and hazard to water: • causes severe skin burns and eye damage • highly hazardous to water (WGK 3)
Lithium cobalt dioxide (LCO, $LiCoO_2$) 	At high temperatures, exothermic decomposition reaction with release of oxygen Toxicity and hazard to water: • irritant, hazardous to health • Cobalt salts can lead to cardiomyopathy (heart muscle disease) • possibly carcinogenic
Lithium-nickel-manganese-cobalt oxide (NMC, $LiNi_xCo_yMnzO_2$) 	Toxicity and hazard to water: • toxic, Co, Ni in the compound • Hazard to water: no information
Lithiumtitanate (LTO, $Li_4Ti_5O_{12}$) 	Toxicity and hazard to water: • slightly irritating to the respiratory tract • Nanocrystals can be toxic due to their small size

Overall, Table 2 and Table 3 summarize the components within LIBs that contribute to environmental and human health risks. In this context, it becomes clear that LIB recycling must understand and consider these risks at all parts of the reverse chain methodology used to recover precious material resources. This results in difficult intermediate storage, collection, and recycling processes specifically developed for LIBs. In all these recycling steps, LIBs must be considered critical devices in terms of spontaneous combustion, hazardous chain reactions and release of eco- and human-toxic gases - regardless of their SOC. The full understanding of the risks associated to LIBs must be supported by their adequate identification and evaluation through standardized safety tests, as discussed in the next section.

2.1.2 The LIB recycling process

Considering the safety and environmental risks, the current recycling process adopted at industrial scale is a multi-stage complex process chain. The LIB recycling technologies can be divided into different steps, *i.e.*: 1) pre-preparation, 2) pre-treatment (including thermal and mechanical Pre-treatment), 3) pyrometallurgy and 4) hydrometallurgy (Figure 1). All industrial available technologies have in common that the recovered materials must undergo a final hydrometallurgical refining to obtain tradeable and reusable products.

2.1.2.1 Pre-preparation

This optional step includes sorting, disassembling/dismantling and discharging. The dismantling is commonly used for large stationary or automotive battery packs or modules. Typically, for this process step, electrical and other components are separated.

2.1.2.2 Pre-treatment

During the pre-treatment process, the LIBs are chemically and/or physically changed. This process can comprise mechanical or thermal treatments, or a combination of them. This process step is necessary to enable massive recycling (more than 10000 tons/year), avoiding disruptive events in the later recycling process. Thanks to this step, operational disruptions in converters and smelter, *e.g.*, fire, (gas) explosions, and presence of impurities in final products can be avoided.

2.1.2.3 Main treatment: Pyrometallurgy

The materials must undergo a final hydrometallurgical refining. However, an intermediate pyrometallurgical purification can be applied to the battery residues. By this step, unwanted components can be removed, and a unification of the target components can be obtained. This step can compensate fluctuations in the composition of the feedstock of the subsequent hydrometallurgical process, which is extremely sensitive to feedstock composition. Furthermore, battery components like fluorine, graphite, phosphorus (included in Level-2 sensor) can be slagged as they tend to technically disturb the subsequent hydrometallurgy step. In addition, low-value, non-precious metals such as iron (included in Level-2 sensor), manganese or aluminium are slagged. This is currently mainly an economic rather than technical decision, as these materials would cause high material and energy costs through dissolving, precipitation, and filtering by means of the hydrometallurgy process.

2.1.2.4 Main treatment: Hydrometallurgy

The hydrometallurgical refining is highly complex and consists of autoclave loading of the reactants, followed by (chlorine) acid leaching and precipitation and filtering of non-noble metals or undesirable elements. Lastly, the process continues with solvent extraction and nickel electrowinning, ion exchange and cobalt electrowinning. Further roasting, dissolving, precipitation, filtering, and electro-extraction processes take place in parallel and sequentially on side routes to extract copper and other by-metals such as lead and precious metals (4).

Noteworthy, process routes that avoid the pyrometallurgy step show technical and economic disadvantages, since highly contaminated intermediate products require additional efforts and costs to remove impurities in the final hydrometallurgical refining. In fact, from an economic perspective, the most important LIB valuable components are Co Ni and Cu. All these components can be effectively recycled after adequate pre-treatment and pyrometallurgical intermediate purification.

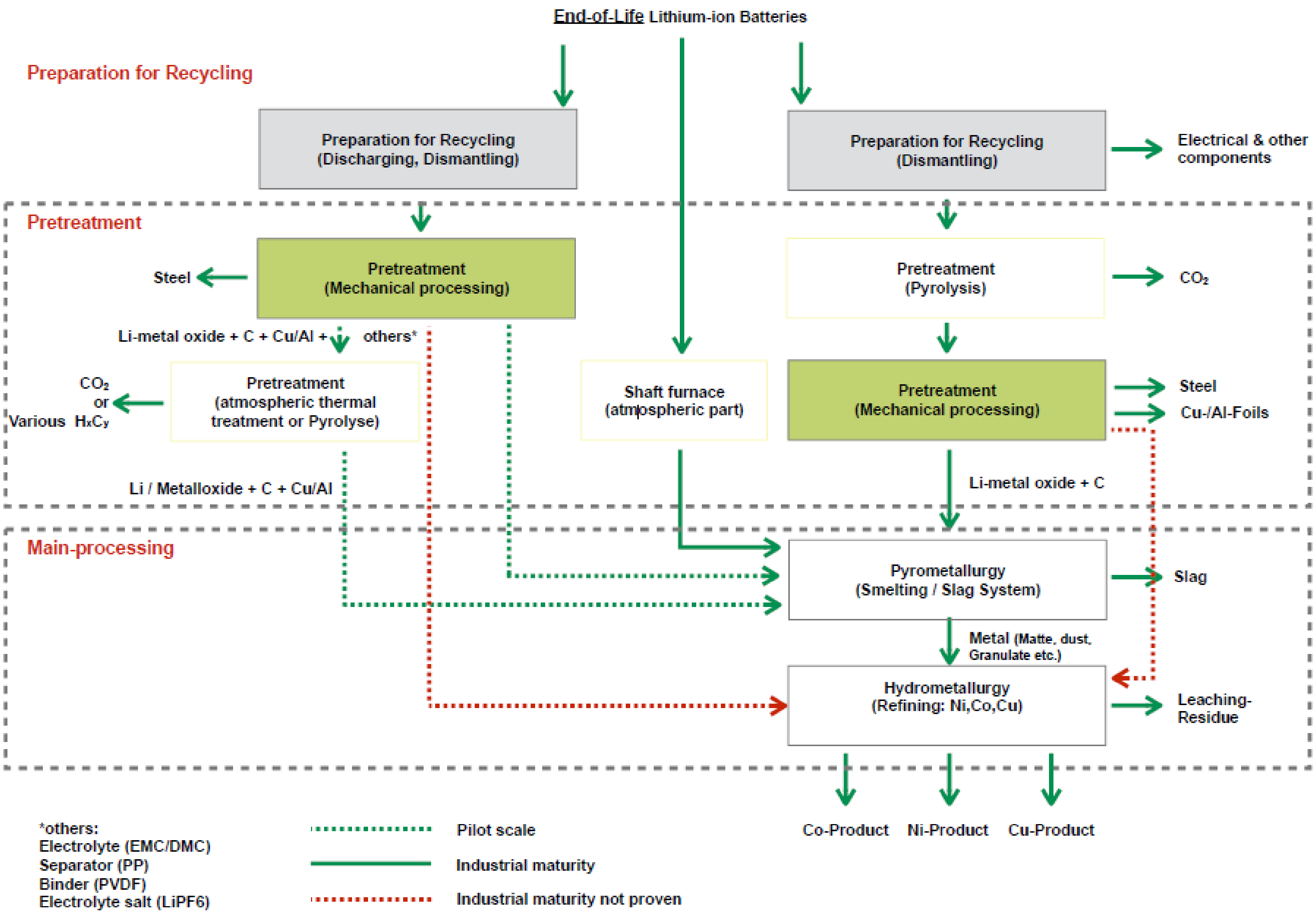


Figure 1: Schematics of LIBs recycling processes (4)

2.1.3 Umicore´s Recycling Solution

To demonstrate the recycling process for LIBs at industrial level, the established process by the company Umicore N.V. will be described in the following. This plant has a recycling capacity of up to 7000 ton/year and is installed at Hoboken, Belgium. The process is mainly composed of smelting in a shaft furnace followed by a subsequent hydrometallurgical treatment.

Smelting process:

The smelting process in the shaft furnace is schematically shown in Figure 2, where LIBs (and NiMH-batteries) and production scraps are directly fed into the smelter without significant preparation treatment at cell level (5,6). Next to batteries, coke, sand and limestone are fed into the smelting furnace as slag formers.

Within the shaft furnace, the temperature is gradually increased. In the upper zone, where temperatures reach ca. 300°C, the electrolytes evaporate. In the middle zone, plastics are pyrolyzed at ca. 700°C. Finally, at temperature of 1200-1450°C, smelting and reduction processes take place at the bottom zone of the shaft furnace (5,6).

The organics (electrolytes and plastics) and graphite (appr. 25-50 wt% of the battery pack) serve as combustible compounds and reducing agent for metal oxides, and the energy released during these reduction reactions provides sufficient energy to heat up the smelter.

By means of a gas cleaning system (UHT technology), the full decomposition of organic compounds is ensured, avoiding harmful dioxins and volatile organic compounds (VOC's). Fluorine is captured by the flue dust.

The resulting metal alloy contains Co, Ni, Cu, Fe and is forwarded to the hydrometallurgical process to recover Co, Ni and Cu. This is carried out after a granulation step, demanded by the leaching step. The resulting slag incorporates, Li, Mn and can be used in the construction industry or further processed to recover valuable metals.

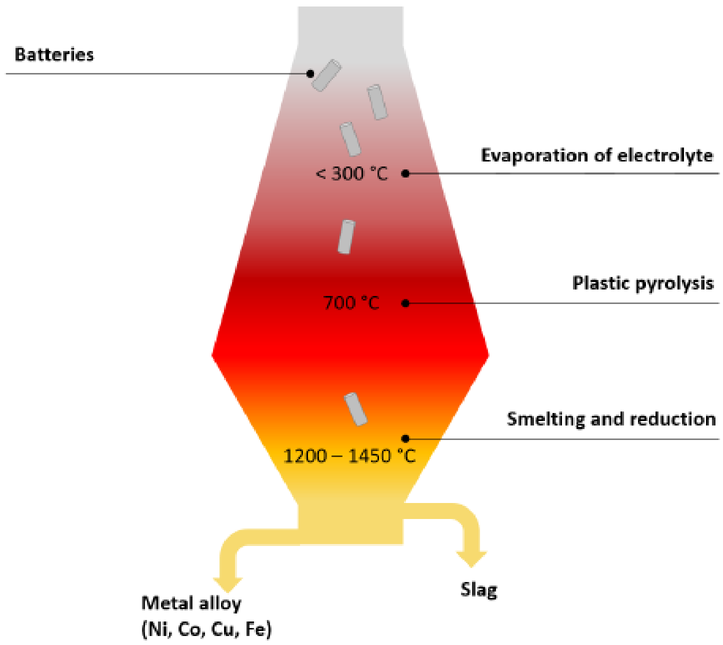


Figure 2: Schematic diagram of Umicore's shaft furnace (6)

The hydrometallurgical refining takes place on the intermediate product derived from the pyrometallurgical treatment. After refinement, different parallel and sequentially processes allow to extract Cu and other non-precious metals. As stated before, Co and Ni must undergo a subsequent refining procedure. The Umicore process scheme is illustrated in Figure 3.

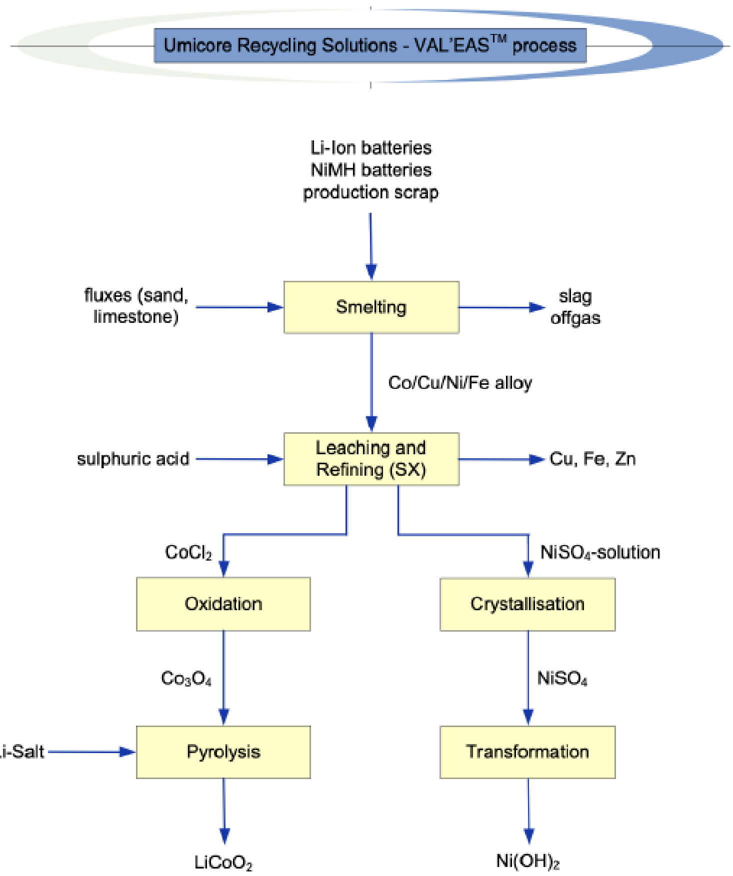


Figure 3: LIB & NiMH battery recycling process published by Umicore (7)

2.2 Recycling Assessment for Level-1 and Level-2 sensor components

Based on the established, industrial recycling processes described in Section 2.1.2 the positioning of the sensor devices (Level-1 and Level-2) inside the cell will impede their disassembling before the pyrometallurgy step. A mechanical pre-processing is industrially incompatible and may also exhibit safety and environmental risks. Table 4 and Table 5 summarizes the components of Level-1 and Level-2 sensors.

Table 4: Components of Level-1 sensors

Components of Level-1 sensors
Pt, Al, Ti
PI, Parylene-C, Silicone Elastomer
SiO_2, Al_2O_3

Table 5: Components of Level-2 sensors

Components of Level-2 sensors
Cu or Al
PVdF or Na-CMC
LFP or LTO
Carbon black
Few-layer graphene
Kapton-tape
PP

A Level-1 sensor matrix in 5 Ah cell format has an overall weight of ~0.50 g, which makes up around 0.5 % of the overall cell weight. Approximately 95% of those 0.50 g polymers like PI, Parylene-C and silicone elastomer, will pyrolyze during the recycling process, providing energy to heat up the smelter (compare UMICORE process; Section 2.1.3). The used ceramics are chemically already present in the slag forming materials. Metal components like Pt and Ti will be recovered via hydrometallurgical and refining routes, while Al is typically received from pre-treatment processes. These metals, however, are only present in trace amounts compared to the overall cell since the sensors are manufactured *via* thin-film processing techniques. To further elaborate this, the amount of Pt present in one 5 Ah Level-1 sensor matrix can be calculated as example. Based on the area of the T-Sensors obtained from the lithography masks (77,6 mm^2), the deposited thickness of the film (50 nm) and the density of platinum (21,45 g/cm^3), the Pt weight is estimated to be 83 µg, which is negligibly compared to the overall cell weight.

For Level-2 sensors (very low mass fractions of 1%), a recycling assessment is straightforward since the sensor is based on components that are typically used to realize the electrodes and the separators of common LIBs.

3 Impact of SENSIBAT sensors on Future Recycling Requirements

The integration of SENSIBAT sensors into lithium-ion batteries holds significant implications for future recycling practices. By providing crucial data on battery performance, usage and health, SENSIBAT sensors enhance the li-ion battery circular economy (Figure 4).

Figure 4: Li-ion battery circular economy (8)

First, the use of L1 and L2 sensors may extend the estimated lifespan of conventional lithium-ion batteries in their EV usage (or 1st life application). Moreover, they enhance the feasibility of second-life battery (2LB) usage for these batteries in less demanding applications like low power electromobility applications or stationary energy storage, thereby reducing the need for immediate recycling and minimizing climate-impacting emissions associated with battery production and recycling.

In addition to their role in extending battery lifespan, SENSIBAT sensors play a vital role in streamlining the repurposing, remanufacturing or recycling process itself. Prior to reusing or recycling, sorting and in some cases disassembling steps are essential, and the information provided by the SENSIBAT sensors can significantly reduce the testing needs in these phases including an obvious mitigation of safety hazards, particularly regarding fire risks—a primary concern for remanufacturing and recycling operators. By offering insights into the historical usage, State of Health (SoH) and State of Safety (SoS) of batteries, the sensors enable more precise sorting and separation of materials based on their condition, optimizing the extraction, refining, and separation of valuable metals from end-of-life batteries.

Furthermore, the SoH data provided by the sensors opens up possibilities for cell remanufacturing approaches. Valuable components, such as the cathode, can potentially be recycled through direct methods. This involves recovering cathode materials from cells without breaking them down and regenerating them through purification or addition of new materials. For instance, eutectic mixtures of lithium iodide and lithium hydroxide can dissolve and regenerate cathode materials at low temperatures, representing a promising technique in battery recycling. Direct recycling methods are emerging as a trend in the field, offering efficient and sustainable solutions for battery recycling challenges.

4 Technical, Economic, Manufacturing and (limited) Environmental Assessment of the Sensor-equipped Cells and Modules

In this section, an individual assessment for the developed sensors-equipped battery cells is summarized.

Following internal discussions between project partners, as well as discussions in the frame of Batteries2030+ network and discussions with the advisory board members, the potential application of the developed sensors shifted compared to initial expectations. Even though a wide application of such sensors by direct integration in the battery assembly process was initially targeted, the project progress revealed that the usage of the developed technologies in R&D activities may be preferable. The application of SENSIBAT's sensors for R&D activities was also supported by the KER assessment of the project outcomes. The planned form of exploitation is mainly internal and for further research programs.

Overall, based on the technical assessment of the project's sensors, the partners foresee mainly two applications:

- R&D departments of battery manufactures may use the developed sensing technologies to gain a more detailed insight in their battery concepts. By doing so, the sensors can provide valuable information on battery performance and helps to reduce costs and time of prototyping activities needed to realize advanced battery configurations.

- Company involved in the design/construction/selling of LIB modules are capable to gain information on individual cell level within the module (e.g., cooling design, performance behaviour under different conditions). Until now, these data are mainly based on modelling results or measured outside the cell.

For these applications, Level-1 and Level-2 sensors represent powerful tools allowing to learn and understand on cell/electrode behaviour, which allows safety-enhancement and increased cycle life in LIBs.

In general, deliverable 3.3 and 3.5 revealed the electrochemical behaviour of cells integrating Level-1 and Level-2 sensors. The most important result of these deliverables and the related tasks was the circumstance that the sensors do not change the cell behaviour (see Figure 5 and Figure 6, taken from previous deliverables).

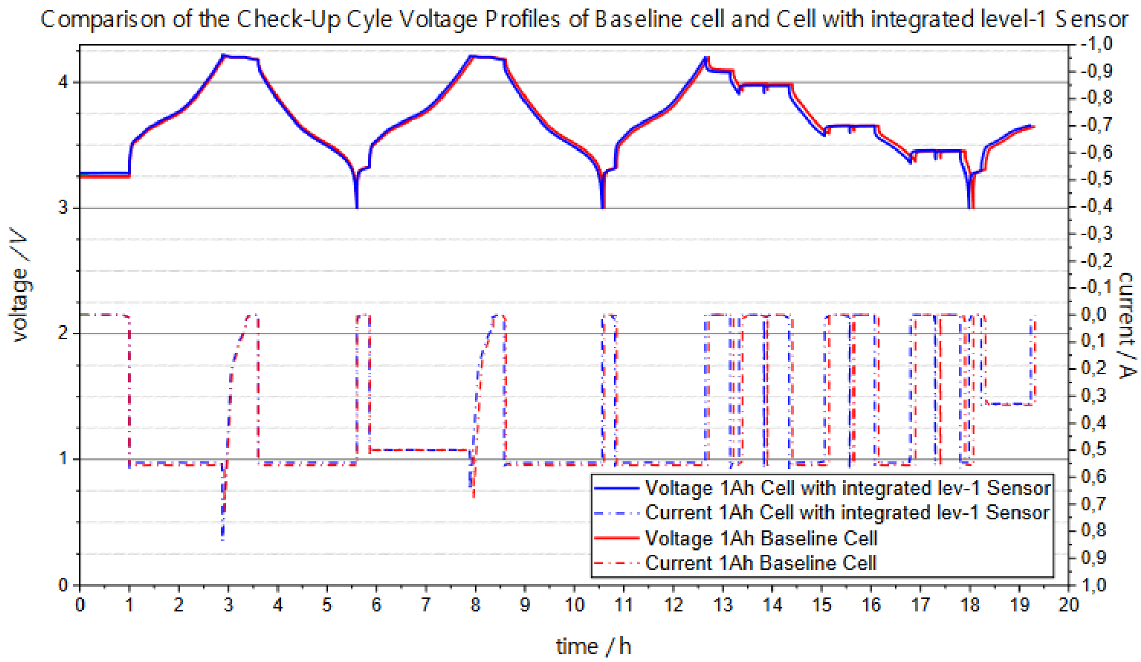


Figure 5: Comparison of the check-up Cycle voltage profiles of baseline cell and cell with integrated Level-1 sensor from deliverable 3.3

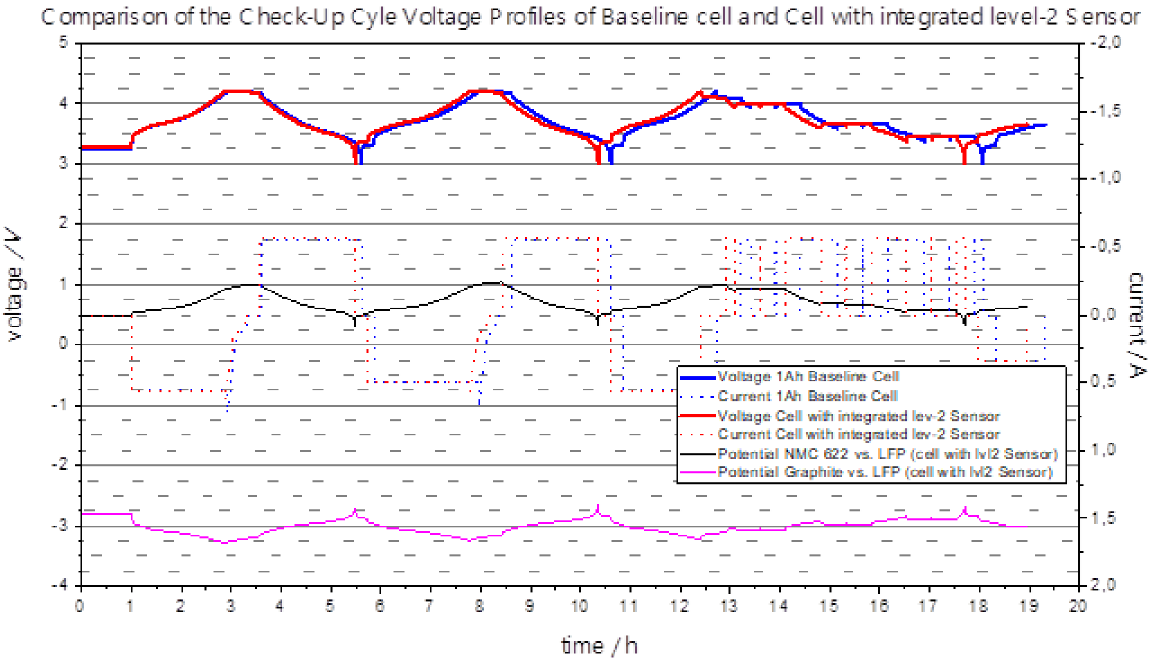


Figure 6: Time-based comparison of check-up tests for an exemplary baseline cell and a Level-2 sensor (un-preconditioned)-integrating cell at VAR (with applied jig) from deliverable 3.5

Economic assessment: For both above-mentioned applications, Level-1 and Level-2 sensors represent a very powerful tool and have the potential for large savings and income, improved sell safety and prolonged cycle life of LIBs. Based on results described in deliverable 5.2, Level-2 sensors don't increase the overall cost of the cells considerably, while Level-1 sensors are too expensive for wide-spread application. However, for R&D-related aspects, *e.g.*, quantification of mechanical stress associated with novel active materials, pressure rise caused by abuse conditions (e.g., elevate temperature), Level-1 sensors still represent a powerful and affordable R&D tool.

Furthermore, when considering economic (and environmental aspects), the precise measurement and estimation of SOHs and SOSs enabled by the sensors will have indirect benefit on cycle life, calendar life and safety of LIBs. This will indirectly contribute to economical and ecological goals for energy storage devices.

Manufacturing assessment: acquiring information on processes taking place inside the cell demands additional feedthroughs to transfer the information outside the cell, leading to unpractical manufacturing routes. For the case of Level-1 sensors, less measurement spots should be considered, as it would simply the feedthrough manufacturing. However, this approach would limit the areal resolution of the Level-1 sensors. Level-2 sensors of the developed design, demand an additional separator layer, which must be considered in the design of automated battery pilot lines.

Environmental assessment: for R&D activities, the use of SENSIBAT's sensor has strongly positive environmental impact. The sensors will provide additional information on the SOHs and SOSs, which can be used to identify optimal operating conditions for LIBs, and thus adequate end-applications. As described before, this will help to increase the lifetime and safety of the cells, connected to environmental and economic benefits. Both sensors will not influence the nowadays established recycling routines, as the components and elements are widely ingredients of LIBs.

5 Discussion & Conclusion

The results presented in this document show that the objective of Task 5.3 was achieved. It comprises an assessment based on the electrochemical results obtained within Del. 3.3 – 3.5 and Del 5.2. The partners have realized 1 Ah and 5 Ah pouch cells with Level-1 and Level-2 sensors, as well as the read-out of such sensors in a 6x5Ah pouch cell module. The results could prove that the integration of the sensors had a negligible or non-detectable impact on the cell performance.

Within the present deliverable a recycling assessment was performed for such cells. Regarding the incorporated sensors, no negative impact could be detected on recyclability.

The deliverable concludes with a technical, economic, manufacturing and environmental assessment. By this conclusion the project partners propose potential user cases for the developed technology. In general, the developments will rather contribute during R&D of cell materials and module design, then be industrially produced in large quantities. Anyhow, this is not necessarily required to provide huge impact on future cell/electrode design, SoH observation, a deeper understanding of LIB aging processes and safety.

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