



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

GA 957273

D3.2 – REPORT ON PROTOTYPING BASELINE POUCH BATTERY CELLS

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Summary

This deliverable D3.2 “Report on prototyping baseline pouch battery cells” summarizes the activities related to the Task 3.2 of Work Package 3. The main objective of this task is to produce standard prototype pouch cells of 1Ah and 5Ah capacity. The cells fabricated in this task will serve as a baseline/reference cells to compare with the cells integrated with Level 1 (1 Ah and 5 Ah cells) and Level 2 sensors (1 Ah cells). To fabricate the baseline cells VAR will prepare and share state of the art standard electrodes (graphite anode and NMC622 cathode) sheets with the project partners ABEE and AIT for the manufacturing of the baseline cells. All the pouch cells will be fabricated with stacked design i.e., anode/ separator/cathode layers will be stacked as many times as needed and connected in parallel to reach the overall electrode area and target cell capacity. Thus, fabricated cells will undergo a formation cycle at C/10 rate in room temperature, 25 °C.

For the benefit of the project few re-arrangements have been done on cells fabricated in each cell format, without reducing the final number of cells.

This deliverable and the related task do not include any deviation from the objectives and timings planned in the Grant Agreement of the SENSIBAT project.



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Abbreviations

Symbol / Abbreviation	
LIBs	<i>Lithium Ion Batteries</i>
CB	<i>Carbon Black</i>
NMP	<i>N-Methyl-2-pyrrolidone</i>
PVDF	<i>Polyvinylidene difluoride</i>
WP	<i>Work Package</i>
DOW	<i>Description of Work</i>
EV	<i>Electric Vehicles</i>
EC	<i>Ethyl Carbonate</i>
DMC	<i>Dimethyl Carbonate</i>
VC	<i>Vinyl Carbonate</i>
FEC	<i>FluroEthylene Carbonate</i>
NMC622	<i>LiNi_{0.6}Mn_{0.2}Co_{0.2}O₂</i>
EIS	<i>Electrochemical Impedance Spectroscopy</i>



1 Introduction

SENSIBAT's starting point is the fabrication of baseline Lithium Ion Batteries (LIBs) consisting of NMC 622 cathodes, graphite anodes, and a standard liquid electrolyte in pouch cell format. NMC 622 is chosen, because it is expected to be the battery chemistry with the highest demand worldwide for the coming 5 years in automotive applications. Graphite is chosen, since it is the key component that has paved the way for the success of LIBs in the past 30 years. Pouch cells have been chosen, because most EV- manufacturers prefer pouch cells rather than the traditional cylindrical or prismatic cells. The advantage of pouch cells is their much higher surface-to-volume ratio for a given capacity, which for example permits better cooling.

The idea behind the fabrication of baseline cells, without integrated sensor, is to compare and study the electrochemical performance and safety of cells comparing them with integrated Level 1 and Level 2 sensors.

According to the Description of Work (DOW) forty 1 Ah and twenty 5 Ah baseline cells must be fabricated for comparing with the twenty 1 Ah Level 1 sensor integrated cells, twenty 1Ah Level 2 sensor integrated cells and twenty 5Ah Level 1 sensor integrated cells, respectively (Table 1). Certain minor re-arrangements have been made in the number of cells in each format (without changing the total number of cells) to benefit the goal of the project (Table 2).

These rearrangements allow the partners achieve higher measurement precision, as VAR and ABEE would produce cells by hand stacking, while AIT can use their automated stacking line. This allows higher measurement precision. Furthermore, the new test matrix allows higher cross comparability of 1Ah cells (baseline and cells with sensors), as the partners do not have the exact same format and cell setup.

The following tables (Table 1 and Table 2) describe the changes made.

Table 1 Cell matrix according to DOW

Partner/Cell Type	Base Line cell	Cell with Level 1 Sensor	Cell with Level 2 Sensor	Total
VAR – 5Ah Cell	20	20	-	40
ABEE – 1Ah Cell	40	-	-	40
AIT – 1 Ah Cell	-	20	20	40

Table 2 New cell matrix agreed by partners

Partner/Cell Type	Base Line cell	Cell with Level 1 Sensor	Cell with Level 2 Sensor	Total
AIT – 5Ah Cell	20	20	-	40
ABEE – 1Ah Cell	10	15	15	40
VAR – 1Ah Cell	10	15	15	40



2 Cell Design and Setup

2.1 Different Cell Formats

Baseline cells of 1 Ah and 5 Ah capacity will be assembled with the aim to prepare the manufacturing of the sensor integrated prototype cells of similar capacity.

As there are three partners responsible for the manufacturing of these cells, the design/dimension of the cell is determined based upon the equipment available for each partner.

Regarding the components used in the cells, VAR provides the needed cathode NMC 622 and anode graphite sheets to all partners, apart from this all the other components are chosen by the respective partners based upon their cell design. Table 3 provides information of the properties of cathode and anode electrode, same for all partners. Table 4 gathers information of the properties of different components used by different partners.

Table 3 Properties of Cathode and Anode electrode Sheets

Electrode	Electrode Total thickness	Current Collector thickness	Electrode Material thickness	current collector weight	Total Electrode weight	Electrode mass loading / Deposition	Electrode Density	Active material
	(μm)	(μm)	(μm)	(mg/cm^2)	(mg/cm^2)	(mg/cm^2)	(g/cm^3)	(mg/cm^2)
Cathode Single Sided	72	15	57	4	22.4	18.4	3.2	17.,7
Cathode double Sided	130	15	115	4	39.6	35.6	3.1	34.2
Anode Single Sided	65	10	55	9	18.2	9.2	1.7	8.1
Anode Double Sided	122	10	112	9	27.4	18.4	1.6	16.1

Table 4 Properties of different components used in pouch cell by partners

Partner/Material	ABEE	VAR	AIT
Electrolyte	1M LiPF6 in EC:DMC v/v 1:1 + 2wt.% VC + 10wt.% FEC	1M LiPF6 in EC:EMC v/v 1:2 +2% VC	1M LiPF6 in EC:EMC v/v 1:2 +2% VC
Separator	Celgard 2500	W-Scope (17.5 μm , 75x55mm))	Celgard 2325
Ni Tab	Length : 60mm Width : 08mm Thickness : 0.1mm	Length : 50mm Width : 4mm Thickness : 0.1mm	Length : 50mm Width : 8mm Thickness : 0.1mm
Al Tab	Length : 60mm Width : 08mm Thickness : 0.1mm	Length : 50mm Width : 4mm Thickness : 0.1mm	Length : 50mm Width : 8mm Thickness : 0.1mm
Pouch Foil	Thickness : 111 μm ±11 μm	Thickness : 153 μm	Thickness : 150 μm

Both the 1 Ah and 5 Ah pouch cells are fabricated by stacking of electrodes and separators as shown in Figure 1. LIB pouch cells made from stacked sheets/electrodes offer much greater safety than conventionally manufactured batteries as the separator shrinks less during battery operation. Thus, short circuits can be avoided in the peripheral areas of a single cell and the safety of the whole battery is increased¹.

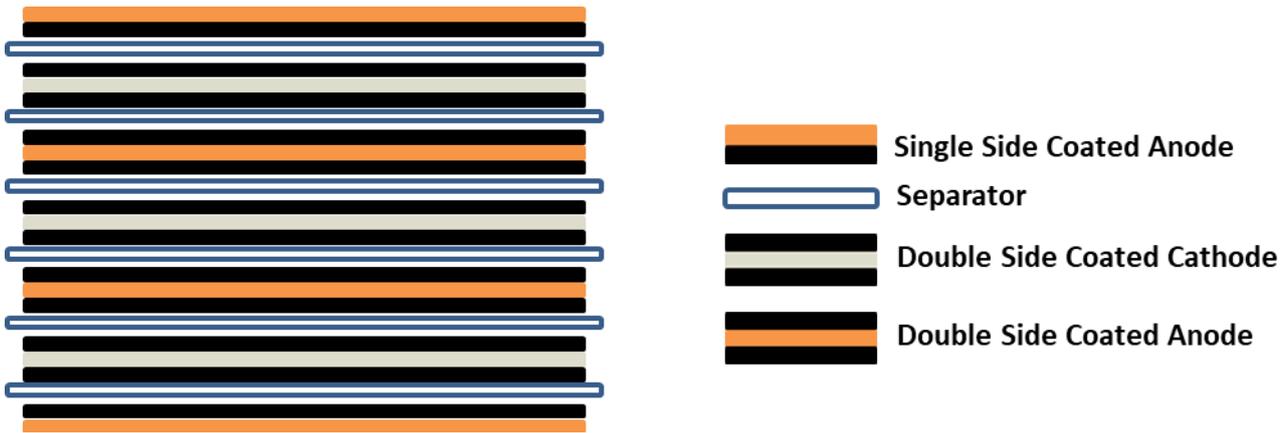


Figure 1 Schematic representation of stacking of electrodes and separators for fabrication of 1 Ah and 5 Ah batteries

2.1.1 Baseline 1Ah Cell

Among the total of 20 baseline 1 Ah cells, 10 cells are fabricated by ABEE and 10 cells are fabricated by VAR. The dimension of the cells varies slightly based upon the fabrication instruments available by each partner. Figure 2 depicts the dimensions of the cells fabricated by ABEE and Figure 3 depicts the dimensions of the cells fabricated by VAR. Table 5 gathers the information of the properties of electrode fabricated by ABEE and VAR.

Table 5. Cell Design Information's for 1 Ah Cell

Properties	Unit	ABEE	VAR
Electrode Dimensions	mm	57*	65*
	mm	44*	45*
Electrode Area one side	cm ²	25	29
Electrode Area both sides	cm ²	50.2	58.5
Capacity (Ah) @Loading	mAh/cm ²	2.7	2.7
Cathode thickness	µm	130	130
Anode thickness	µm	137	137
Nº Cathodes	layers	8	7
Nº Anode	layers	9	8
Nº Electrodes	total layer	17	15
Stack Thickness*(mm)	mm	2.68	2.02

* cathode dimension

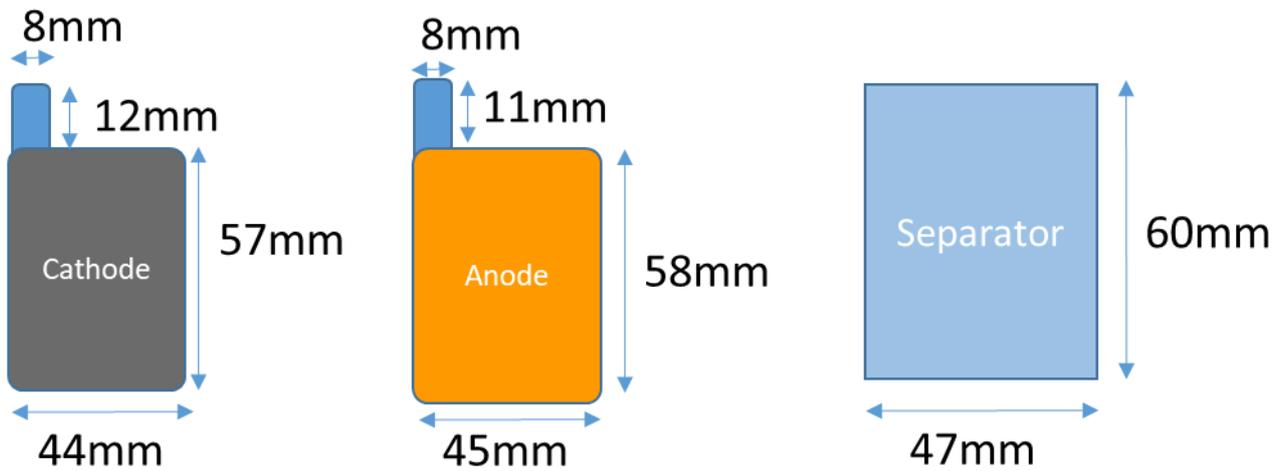


Figure 2 Dimensions of 1 Ah cell fabricated by ABEE

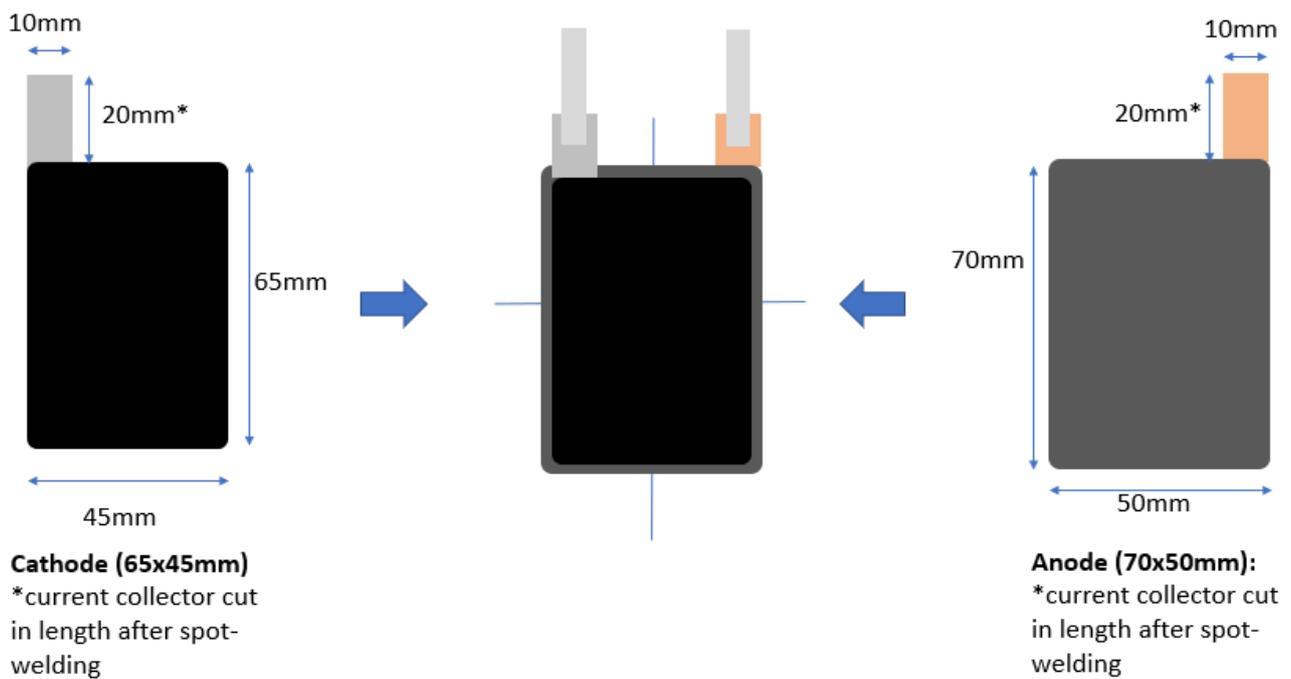


Figure 3 Dimensions of 1 Ah cell fabricated by VAR



2.1.2 Baseline 5Ah Cell

AIT will fabricate 20 baseline 5 Ah cells. The dimensions and properties of the cells fabricated by AIT is shown in Figure 4 and Table 6.

Table 6. Cell Design Information's for 5 Ah Cell

Properties	Unit	AIT
Electrode Dimensions	mm	98*
	mm	68*
Electrode Area one side	cm ²	66.64
Electrode Area both sides	cm ²	133.28
Capacity (Ah) @Loading	mAh/cm ²	2.7
Cathode thickness	μm	130
Anode thickness	μm	137
N ^o Cathodes	layers	13
N ^o Anode	layers	14
N ^o Electrodes	total layer	27
Stack Thickness* (mm)	mm	3.9

* cathode dimension

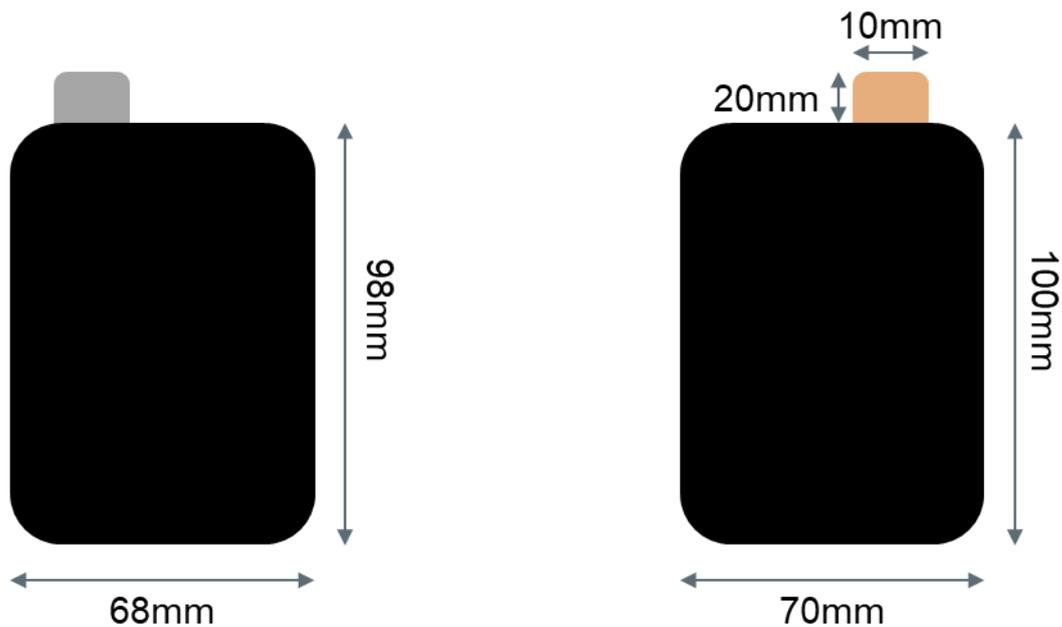


Figure 4 Dimensions of 5 Ah cell fabricated by AIT



3 Component Processing and Cell Assembly

3.1 Cathode electrode Formulation

After drying at 120 °C for 2 hours, active material (NMC622 powders) and Carbon Black (CB) were premixed manually in a mixer, then a part of 2 wt.% polyvinylidene fluoride binder / N-Methyl pyrrolidone (NMP) solution was dropped in it and kneaded. After the mixture became a dough-form, the rest PVDF-NMP solution was added. The mass ratio of NMC622, CB, and PVDF was 96:2:2. This final slurry was coated on a 15- μm -thick aluminium current collector through doctor blade and dried. The electrode was calendared at enhanced temperature and its thickness was reduced to a density of $\sim 3.1 \text{ g/cm}^3$.

3.2 Anode Electrode Formulation

Na-CMC binder and SBR latex binder were mixed with water, followed by the addition of Carbon additives. This process steps were performed using a dissolver mixer. Then a part of the solution was added to graphite and after the mixture became a dough-form, the rest of the solution was added. The ratios of the electrode paste were 87.5% graphite, 8% carbon additives, 1.2 % Na-CMC and 3.3% SBR latex binder. The electrode paste was coated on a 10 μm -thick copper current collector by doctor blade method. The electrode was calendared and the thickness was reduced to a density of $\sim 1.7 \text{ g/cm}^3$.

3.3 Assembly of 1 Ah Cell

In ABEE and VAR, fabrication of 1 Ah pouch cell is carried out inside the glove box (with less than 0.1 PPM O_2) using the procedure described below in flow chart.

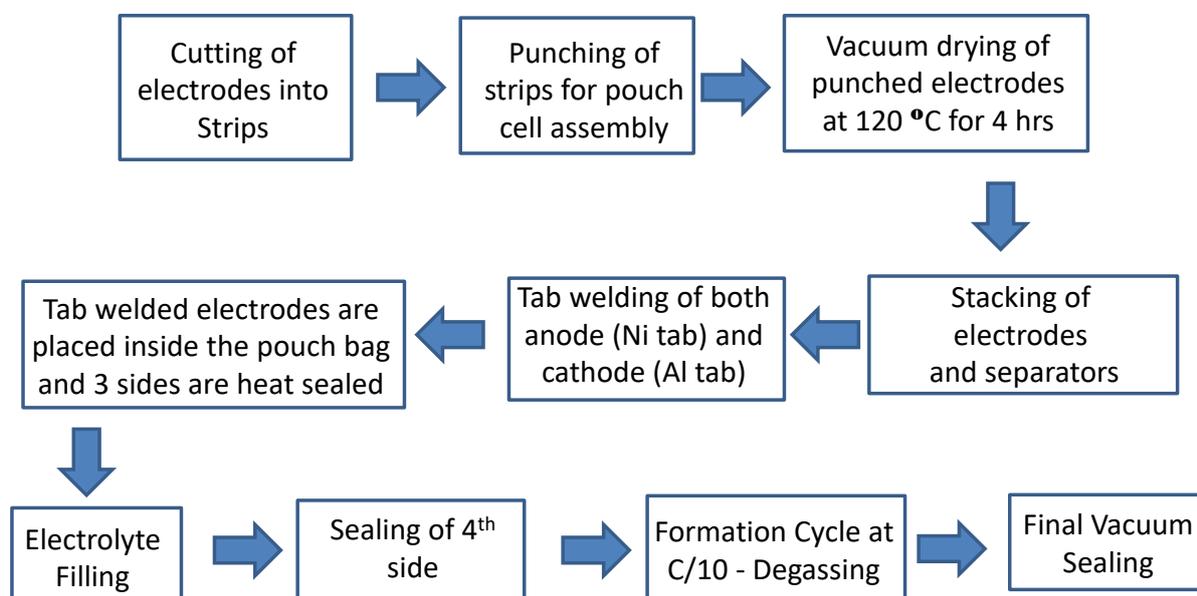


Figure 5. Fabrication of 1 Ah pouch cell

At VAR the cell production is done comparable, but the cells are transferred to the glovebox before the electrolyte filling. In front of this transfer to the glovebox, the cells are dried at 80 °C for 24 hours.



3.4 Assembly of 5 Ah Cell

The assembly of 5 Ah cells is carried out in the same sequence as that of 1 Ah cells in order to ensure a level of comparability (see Figure 5). The stacking process happens using a single sheet stacking machine. Filling of the electrolyte was done under Ar atmosphere and sealing under vacuum.



4 Formation & Check-up Cycles

The formation (preconditioning) process for LIBs typically takes several days or more, and it is necessary for providing a stable solid electrolyte interphase on the anode (at low potentials vs. Li/Li⁺) for preventing irreversible consumption of electrolyte and lithium ions². On the cathode an analogous layer known as the cathode electrolyte interphase layer forms at the high potentials vs. Li/Li⁺.³ The protocol described in Table 7 describes the formation cycle procedure for both 1 Ah and 5 Ah cells. The protocols for the formation and the check-up cycles follow the regime agreed in D1.2 and can also be found in this document.

Table 7. Followed protocol for formation (preconditioning)

Preconditioning													
Formation steps + 30% SOC charging													
Step	Type	Mode	Val	Limit	Val	End Type	Op	Val	Goto	Rpt Time	Val	Options	Step note
1	Rest					Step time	=	24:00:00	002	Step time	00:10:00	ANNN	
2	Charge	Current	0.1C			Voltage	>=	4.2V	003	Step time	00:10:00	ANNN	Formation charge
						StepTime	=	15:00:00	013	Voltage	0.02		
3	Rest					Step time	=	00:15:00	004	Step time	00:10:00	ANNN	
										Voltage	0.02		
4	Discharge	Current	0.1C			Voltage	<=	3.0V	005	Step time	00:10:00	ANNN	Formation discharge
						StepTime	=	15:00:00	013	Voltage	0.02		
5	Rest					Step time	=	00:15:00	006	Step time	00:10:00	ANNN	
										Voltage	0.02		
6	Do1												2 Cycles
7	Advance cycle												
8	Charge	Current	0.1C			Voltage	>=	4.2V	009	Step time	00:10:00	ANNN	
						StepTime	=	15:00:00	013	Voltage	0.02		
9	Rest					Step time	=	00:15:00	010	Step time	00:10:00	ANNN	
										Voltage	0.02		
10	Discharge	Current	0.1C			Voltage	<=	3.0V	011	Step time	00:10:00	ANNN	
						StepTime	=	15:00:00	013	Voltage	0.02		
11	Rest					Step time	=	00:15:00	012	Step time	00:10:00	ANNN	
										Voltage	0.02		
12	Loop1					Loop Cnt	=	2	013				
13	Charge	Current	0.3C			Voltage	>=	4.2V	014	Step time	00:10:00	ANNN	charge 30% SOC
						StepTime	=	01:00:00	014	Voltage	0.02		
14	End												



Table 8. Followed procedure for Check-up – Tests

Checkup Cycles													
Step	Type	Mode	Val	Limit	Val	End Type	Op	Val	Goto	RptTime	Val	Options	Step note
1	Rest					Step time	=	01:00:00	002	Step time	00:10:00	ANNN	
2	Charge	Current	0.5C			Voltage	>=	4.2V	003	Step time	00:10:00	ANNN	0.5C charge
						StepTime	=	07:00:00	029	Voltage	0.02		
3	Charge	Voltage	4.2			Current	<=	0.05C	004	Step time	00:10:00	ANYN	CCC charging
						StepTime	=	01:00:00	004				
4	Rest					Step time	=	00:15:00	005	Step time	00:10:00	ANNN	
										Voltage	0.02		
5	Discharge	Current	0.5C			Voltage	<=	3.0V	006	Step time	00:10:00	ANNN	discharge
						StepTime	=	07:00:00	029	Voltage	0.02		
6	Rest					Step time	=	00:15:00	007	Step time	00:10:00	ANNN	
										Voltage	0.02		
7	Charge	Current	0.5C			Voltage	>=	4.2V	008	Step time	00:10:00	ANNN	0.5C charge
						StepTime	=	07:00:00	029	Voltage	0.02		
8	Charge	Voltage	4.2			Current	<=	0.05C	009	Step time	00:10:00	ANYN	CCC charging
						StepTime	=	01:00:00	009				
9	Rest					Step time	=	00:15:00	010	Step time	00:10:00	ANNN	
										Voltage	0.02		
10	Discharge	Current	0.5C			Voltage	<=	3.0V	011	Step time	00:10:00	ANNN	discharge
						StepTime	=	07:00:00	029	Voltage	0.02		
11	Rest					Step time	=	00:15:00	012	Step time	00:10:00	ANNN	
										Voltage	0.02		
12	Charge	Current	0.5C			Voltage	>=	4.2V	013	Step time	00:10:00	ANNN	0.5C charge
						Current	<=	0.05C	013	Voltage	0.02		
						StepTime	=	03:00:00	013	SetVar	atEnd: V		set Cr
13	Rest					Step time	=	00:30:00	014	Step time	00:01:00	ANNN	Rest 30 min
										Voltage	0.02		
14	Discharge	Current	0.5C			User Def	:	Capacity >=0.1*Var	015	Step time	00:02:00	ANNN	discharge to 90 %
						Voltage	<=	3.0V	015	Voltage	0.02		
15	Rest					Step time	=	00:30:00	016	Step time	00:01:00	ANYN	Rest 30 min
										Voltage	0.02		
16	Discharge	Current	0.5C			Step time	=	00:00:30	017	Step time	00:00:01	ANYN	30sec discharge
										Voltage	0.01		
17	Rest					Step time	=	00:30:00	018	Step time	00:01:00	ANYN	Rest 30 min
										Voltage	0.02		
18	Discharge	Current	0.5C			User Def	:	Capacity >=0.5*Var	019	Step time	00:02:00	ANYN	discharge to 50 %
						Voltage	<=	3.0V	019	Voltage	0.02		
19	Rest					Step time	=	00:30:00	020	Step time	00:01:00	ANYN	Rest 30 min
										Voltage	0.02		
20	Discharge	Current	0.5C			Step time	=	00:00:30	021	Step time	00:00:01	ANYN	30sec discharge
										Voltage	0.01		
21	Rest					Step time	=	00:30:00	022	Step time	00:01:00	ANYN	Rest 30 min
										Voltage	0.02		
22	Discharge	Current	0.5C			User Def	:	Capacity >=0.9*Var	023	Step time	00:02:00	ANYN	discharge to 10 %
						Voltage	<=	3.0V	023	Voltage	0.02		
23	Rest					Step time	=	00:30:00	024	Step time	00:01:00	ANYN	Rest 30 min
										Voltage	0.02		
24	Discharge	Current	0.5C			Step time	=	00:00:30	025	Step time	00:00:01	ANYN	30sec discharge
										Voltage	0.01		
25	Rest					Step time	=	00:30:00	026	Step time	00:01:00	ANYN	Rest 30 min
										Voltage	0.02		
26	Discharge	Current	0.5C			Voltage	<=	3.0V	027	Voltage	0.02		Final discharge
27	Rest					Step time	=	00:15:00	028	Step time	00:10:00	ANNN	
										Voltage	0.02		
28	Charge	Current	0.3C			Voltage	>=	4.2V	029	Step time	00:10:00	ANNN	charge 30% SOC
						StepTime	=	01:00:00	029	Voltage	0.02		
29	End												



5 Results Baseline Characterization

5.1 Resulting Baseline Cells

According to the described specification in this document the partners processed first baseline cells. Examples of these cells are displayed in the following pictures.

5.1.1 Baseline 1Ah Cell at VAR

VAR manufactured the first baseline cells. These cells were manufactured according to the description stated above. Before electrochemical experiments were started, pressure plates were applied on the top and bottom of the cells. The four screws of this jig were tightened with $5 \cdot 10^5 \text{N/m}^2$ with an automated screwdriver.



Figure 6. SENSIBAT 1Ah baseline pouch cells produced at VAR



Figure 7. SENSIBAT 1Ah baseline pouch cell produced at VAR with applied jig



5.1.2 Baseline 1Ah Cell at ABEE

ABEE produced the baseline cells according to the description stated above. Before starting the test, fabricated cells were sandwiched on the top and bottom aluminum pressure plates. The plates were tightened by using torque driver with a torque of 3 Nm.



Figure 8. SENSIBAT 1Ah baseline pouch cells produced at ABEE



Figure 9. SENSIBAT 1Ah baseline pouch cell produced at ABEE with applied jig



5.2 Electrochemical Results

5.2.1 Electrochemical Results of 1 Ah Cell at VAR

Initially the cells were preconditioned using the defined formation procedure. The voltage profiles displayed below, show a good comparability of the produced cells. This formation procedure displays that the requested cell capacity of 1 Ah could be achieved.

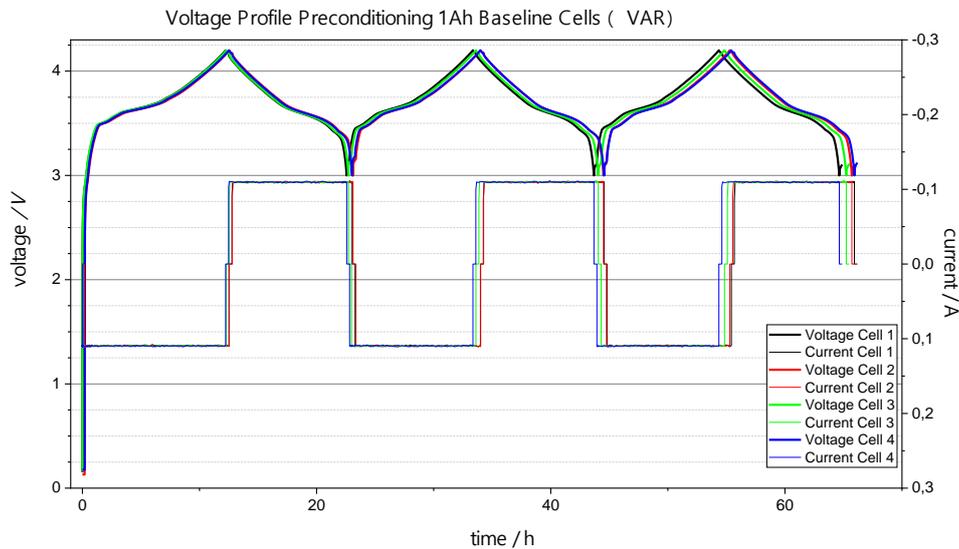


Figure 10. Voltage profile of the SENSIBAT 1Ah baseline pouch cell produced at VAR with applied jig

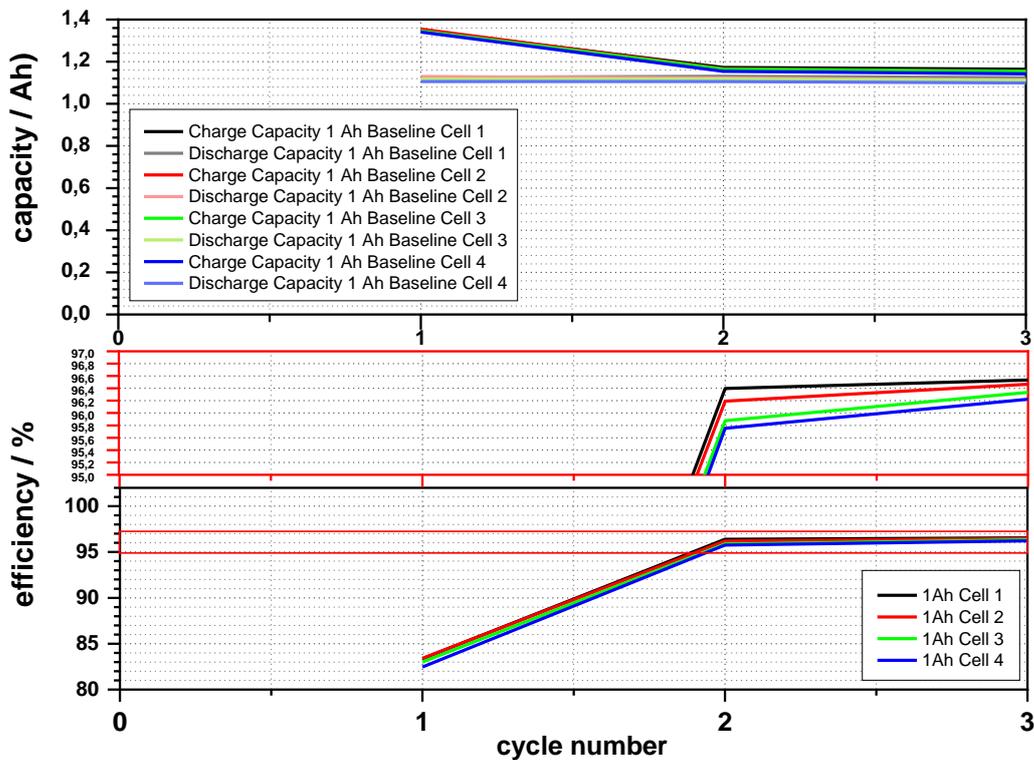


Figure 11. Achievable capacity of the SENSIBAT 1Ah baseline pouch cell produced at VAR with applied jig



In a second step three of the cells were tested according to the Check-up test procedure. The cells could deliver a very comparable performance, which will allow a high comparability with cells that will incorporate sensors.

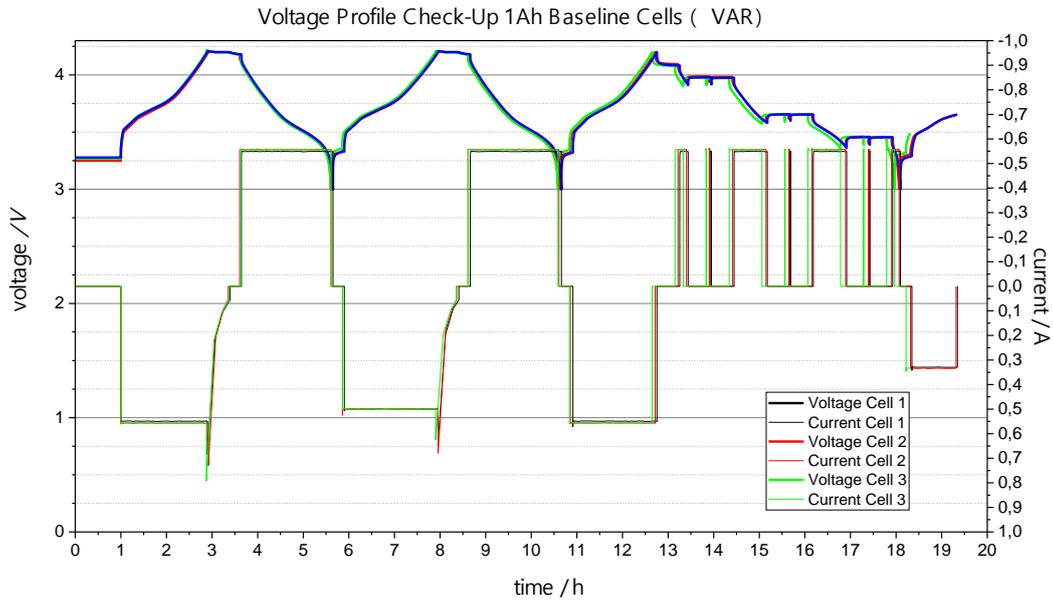


Figure 12. Voltage profile of the SENSIBAT 1 Ah baseline pouch cell produced at VAR (with applied jig) during the check-up test

The 0.5 C pulses at SOC of 90 %, 50 % and 10 % in the Check-up procedure was used to calculate the internal resistance of the cells.

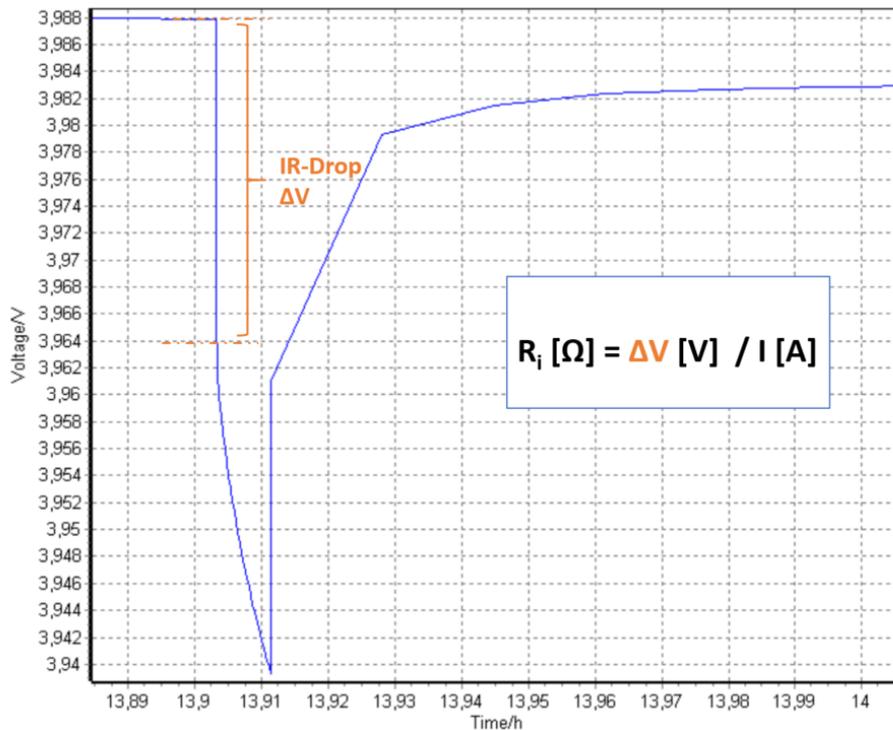


Figure 13. Used IR Drop to calculate the internal resistance



	R _i [mΩ] at 90%SOC	R _i [mΩ] at 50%SOC	R _i [mΩ] at 10%SOC
Cell 1	~50	~60	~60
Cell 2	~70	~70	~70
Cell 3	~130	~140	~140

Table 9. Calculated IR values

The 1 Ah cells at VAR display internal resistances of ~50-70 mΩ. One of the investigated cells displayed a significantly higher internal resistance (marked in yellow).

5.2.2 Electrochemical Results of 1Ah Cell at ABEE

The cells were preconditioned initially using the defined formation procedure as show below. The voltage profiles displayed below, show a good comparability of the produced cells. This formation procedure displays that the requested cell capacity of 1 Ah could be achieved.

Table10. Followed protocol for formation (preconditioning)

Property	Value	Property	Value	Property	Value
Dev_Unit_Ch1	#22_10_8	Volt. Upper	--	Aux Volt. Upper	--
Start Time	2021-04-19 11:17:32	Volt. Lower	--	Aux Volt. Lower	--
Start StepID	1	Cur. Upper	--	Aux Temp. Upper	--
Time in Step	00:03:16:000	Cur. Lower	--	Aux Temp. Lower	--
Capacity	0.0000 Ah	Cap. Upper	--	Aux Volt. Range	--
Cycle	1	Barcode		Aux Temp. Range	--
Record Condition	10 sec 0.0 mV 0.000 mA	P/N	2021-04-19 11-17-32	Aux Cur. Range	--
Volt. Range	5 V	Creator		Aux Record Con...	0 sec 0 mV 0 °C
Cur. Range	±6000mA/100mA/3000mA	Remarks		Aux Diff Volt.	--
Step File		Loop Impedance	--		

ID	Step name	StepTime(hh:mm:ss:ms)	Rate(C)	Volt(V)	Cur.(mA)	Cap.(Ah)	Stop
1	Rest	24:00:00:000					
2	CC_Chg	15:00:00:000	0.100	4.2000	112.9		
3	Rest	00:15:00:000					
4	CC_Dchg	15:00:00:000	0.100	3.0000	112.9		
5	Rest	00:15:00:000					
6	Cycle	Begin ID: 2	Times: 2				
7	End						

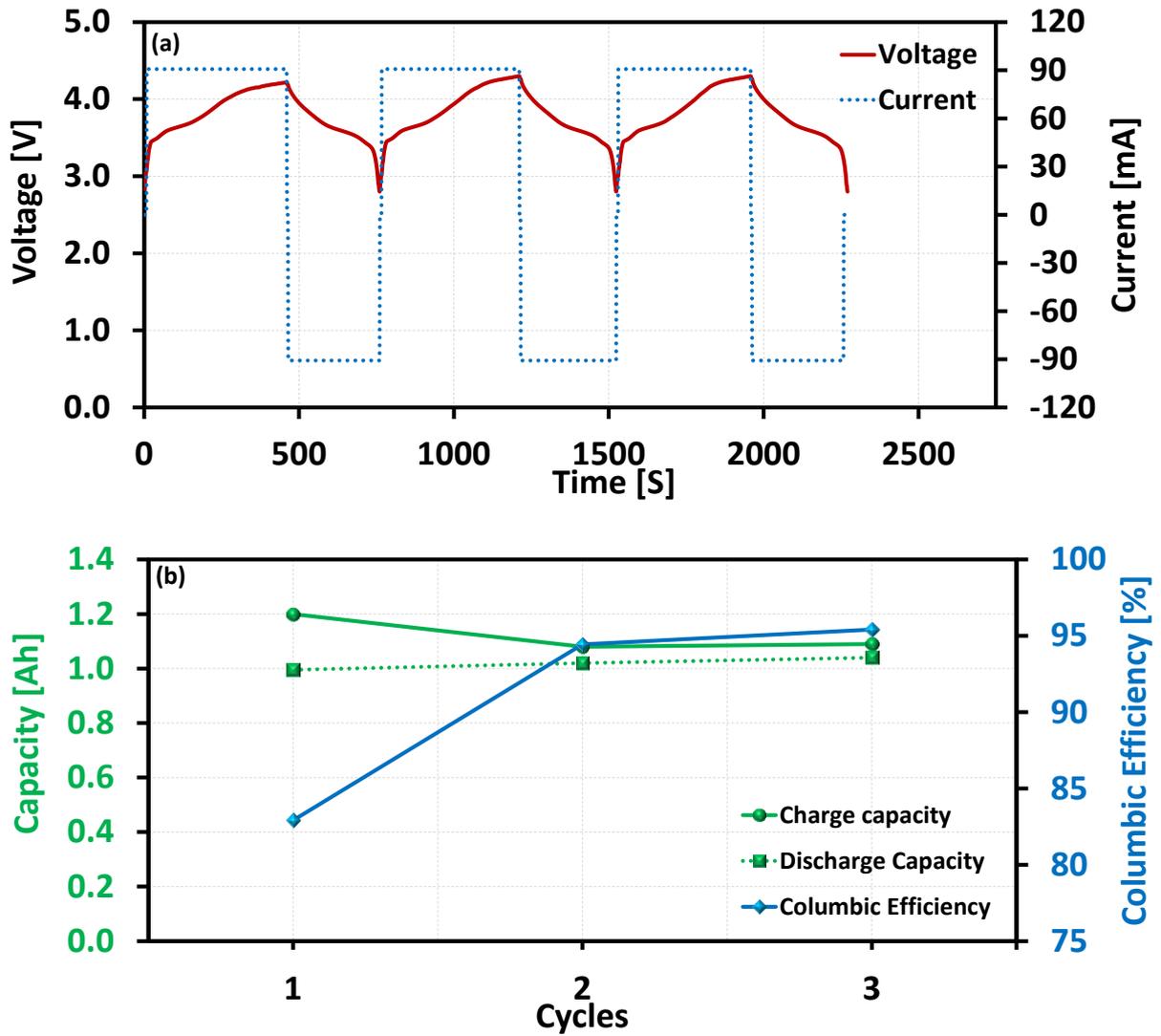


Figure 14. Voltage profile of the SENSIBAT 1Ah baseline pouch cell produced at ABEE with applied jig (a) and formation cycle data with columbic efficiency (b)



6 Discussion & Conclusion

The present deliverable incorporates the manufacturing procedure of cathode (NMC622) and anode (Graphite) electrodes by VAR, fabrication of 1Ah (by ABEE and VAR) and 5 Ah (by AIT) baseline cells, formation cycles and check-up cycles of fabricated baseline cells. Loading of active materials in the cathode and anode electrode during the manufacturing process is specified by VAR and the number of electrodes required to fabricate 1 Ah and 5 Ah cells are specified based on the dimensions of the cells by VAR, ABEE, and AIT, respectively. All the partners used stacking process to fabricate the baseline cells.

First measurements with the baseline cells prepared at ABEE and VAR display a high reproducibility and high comparability. In this context, the baseline cells will allow the partners to clearly display effects triggered by the implementation of Level 1 and Level 2 sensors. Further, these baseline cells will be used to do the other specified analysis in this project like cycle life, calendar life and EIS at room temperature and high temperature (Defined in D1.2).



7 Risks

No risks related to D3.2 have been identified.



8 References

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4	FHG	FRAUNHOFER GESELLSCHAFT ZUR FOERDERUNG DER ANGEWANDTEN FORSCHUNG E.V.	Germany
5	FM	FLANDERS MAKE VZW	Belgium
6	TUE	TECHNISCHE UNIVERSITEIT EINDHOVEN	The Netherlands
7	NXP NL	NXP SEMICONDUCTORS NETHERLANDS BV	The Netherlands
8	NXP FR	NXP SEMICONDUCTORS FRANCE SAS	France
9	ABEE	AVESTA BATTERY & ENERGY ENGINEERING	Belgium
10	VAR	VARTA MICRO INNOVATION GMBH	Germany
11	AIT	AIT AUSTRIAN INSTITUTE OF TECHNOLOGY GMBH	Austria
12	UNR	UNIRESEARCH BV	The Netherlands

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