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List of requirements for the integration of the multisensor platform in cells

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Task	1.2	Definition of Requirements for Integration of Sensors into the Cells	

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Deliverable abstract

This report contains the requirements and restrictions for the integration of a multi-sensor platform into a lithium-ion cell. These restrictions and requirements refer to the consequences for the lithium-ion cell on the one hand and to those for the integrated sensors on the other. Methodically, a TRL5 is assumed for the prototype cells designed in the project and a TRL4 is aimed for after integration of the multi-sensor platform. For the respective technical maturity levels of the INSTABAT prototypes, important key performance parameters are defined and compared with key performance parameters of a virtual state of the art (SoA) lithium-ion cell with a TRL9. All specifications given in this document are understood to be tentative and may change depending on the project progress.

Deliverable Review

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Product Requirement Document

Preamble: Any strictly necessary additional requirements that arise in the future will be immediately communicated to the INSTABAT project partners. The specifications for prototype cells and test protocols are based on assumptions and empirical values for comparable cell systems. Adaptations of the test protocols and specifications can be expected according to actual requirements and findings.

1 General

This document contains the preliminary specifications and requirements for the prototype cells to be manufactured and characterized in the scientific work packages within the *INSTABAT* project. Furthermore, the critical aspects and resulting restrictions regarding the integration of a multi-sensor platform into the prototype cells are addressed. The methodological approach is as follows. The sole prototype cell without an integrated sensor or the multi-sensor platform is specified as state of the art *(SoA)*. In contrast, the prototype cell with the integrated multi-sensor platform is specified in a second step. For this, the boundary conditions for the integration and the influences of the sensor integration on the capabilities of the cell must be critically evaluated at this point at the latest. The advantages of the information gained by the sensors must at least outweigh possible losses in cell performance due to their integration. Consequently, the top premise is that the multi-sensor platform shall be adapted to the prototype cell and not the other way around.

State-of-the-art cell chemistry is planned for the projected prototype cell. *Graphite* is used as the negative active material (anode) and *NMC622* is used as the positive active material (cathode). A porous polymer membrane (PP/PE) serves as the separator and a solution of organic carbonates and LiPF₆ as conductive salt is used as the electrolyte. The format of the preliminary prototype is a stacked pouch cell with a capacity of 1.0 Ah to 1.5 Ah. *Table 1* provides the main characteristics on the stack and electrode dimensions.

			thickness	capacity	
		mm x mm	μm	mAh/cm²	
Anode (double-sided)	current collector	52 x 52	10	3.0	
	graphite	52 × 52	120		
Separator PP/PE		54 x 54	25		
Cathodo (doublo-sidod)	current collector	50 x 50	15	27	
	NMC622	50 X 50	104	2.1	

Table 1: Data on stack and electrodes dimension for tentative INSTABAT Prototype Cell

The specifications listed in *Table 1* are preliminary and may change in course of the project according to the latest findings. At a later stage, it is planned to use a commercially available and suitable silicon-carbon composite (<5% Si) as the anode-active material. In order to achieve the greatest possible comparability, preliminary specifications will be developed for a hypothetical SoA cell (*TRL9**), for the INSTABAT prototype base cell (*TRL5***) and the INSTABAT prototype cell with the

integrated sensor platform (*TRL4****)⁴, depending on the level of technical maturity. Even though the main task of the INSTABAT project is to advance the maturity of the "Smart Cell"⁵ technology through experimental research and to open up insights into new findings, all known and, according to the current state of knowledge, challenging restrictions with regard to material selection and electrochemical requirements are openly and clearly addressed and discussed.

2 Cell requirements

2.1 Mechanical requirements

The requirements that can be assigned to the mechanical aspects first concern the dimension of the battery cell and the multi-sensor platform. The dimensions of commercially available battery cells are largely specified by international standards. For a cylindrical cell, e.g. 26650, a height of 64.5 mm \pm 0.2 mm and a diameter of 26.26 mm \pm 0.2 mm would be typical. With these two key figures, the available volume in the cells is defined in a first approximation and the cell manufacturer is faced with the optimization task of utilizing the volume to the maximum in order to achieve maximum capacity and energy density. For the dimensioning of the sensors, one of the main requirements for the multi-sensor platform is to be as small as possible in order to have as little negative impact as possible on the capacity and energy density of the cell. The small size of the sensors also plays an important role in their positioning, which will be discussed below. However, in order to obtain the maximum possible degree of flexibility in the course of the upcoming development work, a stacked cell, with a housing based on a polymer-aluminum composite foil (soft-case/pouch), was chosen for the INSTABAT project. The advantage of this cell is a high degree of variability in the possibilities for changing the stack geometry and the realizable and required positions for integrating the multi-sensor platform.

The specifications regarding the dimensions of the cell and the individual sensor of the multi-sensor platform are provisional and may change during the ongoing project in accordance with the latest findings in consultation with the project partners.

2.1.1 Dimensions / INSTABAT tentative Prototype-Cell

The area dimensions of the preliminary prototype cell are shown in *Figure 1*. The dimensions of the stack with anode, cathode and separator can be found in *Table 1*. The overall footprint of the cell is dominated by the gas- and moisture-tight housing made of a multilayer polymer-aluminum composite foil. The dimensions of the overall footprint are as follows.

- Length = 74 mm \pm 0.5 mm (without tabs)
- Width = 66 mm ± 0.5 mm
- Height = 3.5 mm ± 0.1 mm (BoL)⁶

⁴ For the following chapters, the superscript asterisks indicate the (hypothetical) industrial pouch cell * the INSTABAT prototype cell ** and the INSTABAT Multi-Sensor Platform *** and refer to the initial cell chemistry C/NMC622. All additional stated superscript crosses indicate the advance cell chemistry C/Si₅₅₀⁺⁺/NMC622.

⁵ Joe Fleming, Tazdin Amietszajew, Jerome Charmet, Alexander John Roberts, David Greenwood, Rohit Bhagat, *Journal of Energy Storage*, **2019**, 22, 36-43

⁶ BoL «Begin of Life»

The height of the stack is calculated by adding the number of layers on the anode, cathode and separator necessary for a cell capacity of 1.4 Ah and the thickness of the cell housing.



Figure 1: Footprint for *tentative* INSTABAT Prototype-Cell

For the tentative INSTABAT prototype cell, the width of the weld-seams is 5mm each. The current collector flags for both the anodes and the cathodes are 12m wide and 10mm long. The 4mm wide aluminum-tabs and nickel-tabs are attached to these using a welding process. Of particular importance is that these tabs are coated on both sides with an adhesive polymer to improve metal/polymer adhesion, in order to achieve a reliable, moisture- and gas-impermeable connection at the frontal weld-seam. Analogue solutions must also be applied or necessarily developed for the integration of the sensors for the sensor platform.

2.1.2 Dimensions / INSTABAT tentative Sensor-Platform

The practical dimensions of the individual sensors of the sensor platform are given in a first approximation by the dimensions of the cell. For both smaller cells such as the 21700 and large-format cells such as the wound or stacked PHEVII, the ultimately available space in the cells is tight due to the ubiquitous demand for high energy densities. Nevertheless, ambitious engineers need to face these challenges soberly and start with an assessment of the state of the art, in terms of current size ratios, as well as for the potential for downscaling.

A distinction must be made between the actual **sensor** or **probe** (sensing area), any necessary adaptations of the sensor environment **(support)** and finally the measuring line with which the signal is to be led out of the cell for further processing **(supply line)**. The sum of the masses and volumes of these components can already have a measurable influence on the achievable specific energy and the energy density. Special attention must be paid to the gas-tight feed-through of the measuring line into and out of the cell.

The design and dimensions of the sensor line are to be considered tentative and may change in course of the project.

A detailed example is given using the **luminescent sensors** planned for the INSTABAT multi-sensor platform.

Luminescent sensors are developed on optical fibre by functionalization of the end fibre (see. Figure 2 (right). The left of Figure 2 shows the typical structure of the optical fibre. The core of the fibre is covered by a cladding with a higher optical index. The difference in index constrains the light to propagate into the core. A coating layer above the cladding is deposited to improve the optical properties. Then a strength member and an outer jacket cover the coating to protect the fibre. There are different kinds of fibre with a large choice of dimensions and materials. For this project, we need to take account the optical properties of the fibre for maximizing the sensitivity.



Figure 2 : Schematic view of optical fibre structure and luminescent probe functionalisation at the end of the fibre

An example of attenuation spectra is shown in Figure 3 for two kinds of commercial silica fibre. The optical properties of luminescent probe such as excitation and emission wavelength should be according to the minimum of the attenuation band. This constraint limits the choice for the core and cladding material. The deposition of the luminescent probe for temperature measurement and li-ion concentration requires having a silica core and a cladding easily removable.



Figure 3: Attenuation spectra of two types of silica fibre from Thorslab⁷

⁷ https://www.thorlabs.com/newgrouppage9.cfm?objectgroup_id=1362&pn=FP200ERT

Table 2 lists the specifications from the manufacturer of these two fibres as an example. We can show the wavelength range, the numerical aperture (NA), the optical index of core and clad material, the diameter and materials of core and core with cladding and the materials of coating.

Reference	Wavelength Range (nm)	Hydroxyl content	NA	Core Index @589.3 nm	Clad Index @589.3 nm	Core Diameter	Cladding Diameter	Core/Cladding	Coating
FP200URT	300-1200	High OH	0.5	1.458434	1.3651	200 +- 5	225+- 5	Pure Silica/ Hard polymer	Tefsel
FP200ERT	400-2200	Low OH		1.458965	1.3651	huu	h		

Table 2: Example of specifications of two kind of silica fibres⁷

Table 4 shows a schematic view of the optical fibre insertion into the pouch cell. Only the core with cladding is inserted into the cell. Therefore, we need to consider external diameter of the cladding for the diameter of the probe (cf paragraph 2.1.3). For example from Table 2 if we used this type of fibre, the diameter to be considered is 225 μ m.



Figure 4: Schematic view of the fibre insertion into the pouch cell

The length of the part without outer jacket can be fitted as a function of the position of the probe into the cell. The total length of the fibre could vary according to the manufacturing constraints from centimetres to meters. The end of the fibre is connected to a light source and a detector, both controlled by a computer for measurement and data processing (cf Figure 5).



Figure 5: Schematic view of the fibre connection to the light source, detector and computer for monitoring, data collection and processing.

The size of the light source and detectors is not actually defined because different types of sources will be tested during the project, depending on the materials used for luminescent probe. For the detectors, we will use a portable spectrophotometer or photodiodes associated with optical

components. In any case, all this hardware fits easily in a box around the size of a shoe box. This hardware configuration is the same for the both luminescent sensors for temperature and Li-ion concentration measurement.

Table 3 shows the preliminary dimensions of the components for the planned sensor platform.

Sensor-ID	Sensor type	Measured parameters	Sensor/ Probe length/width+/height		Support length/width*/height		Supply line length/width*/height		ight	Sketch		
			I / mm	w / <i>mm</i>	h / <i>mm</i>	۱/ mm	w / mm	h / <i>mm</i>	L / mm	w / <i>mm</i>	h / <i>mm</i>	
		Temperature	10	0.01	-	10	0.125	-	**	0.125	-	ta fibrica
	Fiber Bragg	Heat Flow	10	0.01	-	10	0.125	-	**	0.125	-	and highly highl
1	Grating (FBG)	Pressure	10	0.01	-	10	0.125	-	**	0.125	-	FBG Sen 00 00 00 00 00
		Strain	10	0.01	-	10	0.125	-	**	0.125	-	Since the second
2	Reference	"absolute" potential	3.0	3.0	0.05	6.0	6.0	0.10	**	3.0	0.03	a mm g Sensor
	electrode	Impedance	3.0	3.0	0.05	6.0	6.0	0.10	**	3.0	0.03	Support Supply line
3	Luminescence	Temperature	1-5	0.1 – 0.25	-		0.12-0.25	-	**	3	-	Inside pouch coll outside pouch rell 125-250 µm 3 mm 50 100 µm
	Luminosochie	Li ⁺ - concentration	1-5	0.1-0.25	-		0.12-0.25	-	**	3	-	^o Probe L'10 mm 3-5 cm 0.2-1 m
4	Photo-acoustic (PA) #	CO ₂ - concentration	(14)	(13.8)	(7.5)	(34)	(28)	(7.5)	(**)	(**)	(**)	NS THE REPORT OF
+ dia # Ini	ameter fiber sensing ar tial approach is to integ	ea * diameter of silio grate the sensor via	ca fiber ** De a microcapil	pends on sen lary tube. Furth	sor positi ner devel	on in the opment c	cell of the sensor, i	n terms of	detection	range and din	nensionir	ng, during the project.

Table 3: Tentative dimension for sensor, support, and supply-line of multi-sensor platform to be integrated into INSTABAT prototype cell

The dimensions of the sensor-line for the multi-sensor platform listed in Table 3 have serious consequences for their planned integration into the INSTABAT prototype cells, which are discussed in the following section.

Exclusion Criteria!

- ! Gas and moisture tightness of the feedthroughs for the multi-sensor platform is not guaranteed.
- ! Reduction of energy density and specific energy by > 15% through integration of the multisensor platform in the INSTABAT prototype cell.
- ! The integration of the Multi-Sensor platform increases the safety hazard level to > 4.8

2.1.3 Location / Integration of sensor-platform into tentative Prototype-Cell

When determining the location for integrating the multi-sensor platform into the cells, the basic questions are what, where, how much and how often should be measured. In answering these according to quantity and sampling rate, the capabilities of the sensors play a major role in the first approximation. The question of what and where quickly leads to the realization that a largely inhomogeneous measurement field will be encountered in a battery cell. Roughly, an arbitrary division can be made here between integration within the stack/coil of the cell and integration in the cell outside the stack. The spatial integration of the multi-sensor outside the stack/coil depends on the available "dead volume" in the cell. In principle, there is no free volume available to integrate a sensor into the stack/coil, but this can be achieved by technical modifications of the stack/coil design. However, when proceeding in this way, it must always be borne in mind that the sensor platform represents a foreign body that is not provided for in the functional principle of the cell and can potentially have serious effects on functionality and safety.

In order to get an idea of the challenges that arise with regard to the sensor integration in the stack, we want to take a closer look at the cross-section of the stack of the tentative INSTABAT prototype cell in Figure 6. This shows a 20-fold magnification of the 3mm high stack in the z-axis.



Figure 6: Cross-section of the stack in z-direction for the preliminary INSTABAT prototype cell

⁸ Hazard Level 4: Venting, ∆ mass ≥50%, no fire or flame; no explosion; weight loss ≥50% of electrolyte weight (electrolyte = solvent + salt)

This makes it easier to see that this stack consists of 10 double-sided coated anodes, 9 doublesided coated cathodes, 20 separators in between and 2 single-sided coated cathodes at the bottom and top. This results in at least 20 possibilities for integrating the sensor or the sensor tip in the zaxis. For the x-axis and the y-axis there are almost infinite possibilities to position the sensor. The positioning of the sensor feed lines required for this must also be considered.

Once an optimal point in the stack for positioning the sensor has been identified, it is worth taking a closer look at the local size ratios at this point. Figure 7 shows the size ratios at one of the possible integration points for the sensor in the z-direction in cross-section. More precisely, it is the area where the electrolyte-impregnated separator with a layer thickness of 25 μ m is located. At this point there would be the possibility to measure the lithium ion concentration and the absolute potential. To get a feeling for the possible size of the sensor tip, cylindrical measuring sensors in y-direction with diameters of 50 μ m, 100 μ m and 150 μ m are shown. From this it is obvious that the maximum possible miniaturization of the sensors or the sensor platform must be one of the goals of the project.



Figure 7: Cross-section in z-direction of a section of the stack for the preliminary INSTABAT prototype cell

Direct contact between the sensor platform and the active materials should be avoided, so sufficient electrical insulation of the sensor tip and sensor leads or the introduction of a second layer of separators at the positions intended for the sensor is recommended. Figure 8 (left) shows a hypothetical integrated cylindrical sensor tip in both the x-direction and the y-direction. In the same figure on the right, a hypothetically integrated reference electrode is shown.



Figure 8: Cross-section in z-direction of a sector for the stack for tentative INSTABAT Prototype Cell; (left) hypothetically integrated cylindrical sensor, (right) hypothetically integrated reference electrode

It should be noted that the separator can be squeezed somewhat due to its porosity and has been taken into account in Figure 8.

Exclusion Criteria!

- ! Sensors with a larger cross-section in the z-direction than 2 times the electrode diameter (~250µm).
- ! Safety-critical deposition of lithium at locations where the sensors are integrated into the stack.
- ! The integration of the Multi-Sensor platform increases the safety hazard level to > 4.8

Furthermore, the multi-sensor platform is also exposed to a mechanically and chemically demanding environment, which must be considered during sensor integration.

2.1.4 Chemical environment

The chemical environment is dominated by the electrolyte and the active materials in the negative and positive electrode. This has far-reaching consequences for the sensor platform. Depending on the measurand and the measuring principle, the multi-sensor platform is exposed to a harsh chemical environment. The electrolyte is initially a mixture of organic carbonates in which a lithium salt is dissolved for the necessary charge transport. The electrolyte components are subject to continuous degradation due to thermal and electrochemical stress, resulting in a constant change of the chemical environment by the electrolyte. *Table 4* shows SoA solvents and the expected decomposition products.

Solvent	Tm/°C	Tb/°C	Decomposition /Ageing
Ethylene carbonate [EC]	+36	248	ethene, Li ₂ CO ₃ , CO ₂ , polyether, polycarbonate
Ethyl methyl carbonate [EMC]	-53	110	alkylcarbonate, alkane, ROLi, ROR, CO2
Dimethyl carbonate [DMC]	5	91	alkylcarbonate, alkane, ROLi, ROR, CO2
Diethyl carbonate [DEC]	-74	126	alkylcarbonate, alkane, ROLi, ROR, CO2
Fluoro ethylene carbonate [FEC]	23	212	ethene, Li ₂ CO ₃ , CO ₂ , LiF
Propylene carbonate [PC]	49	242	propene, Li ₂ CO ₃ , CO ₂ , polyether, polycarbonate
Additive			
Vinylene carbonate [VC]	SEI mo	difier	Polycarbonate
Cyclohexylcarbonate [CHB]	Overcha protec	arge - tion	oligo/poly-CHB, H ₂

Table 4: SoA	solvents and additive	es for Lithium-Ion-Batt	erv electrolytes

One of the essential components of an electrolyte for lithium-ion batteries is the conducting salt. Nowadays, lithium hexafluorophosphate (LiPF₆) is almost exclusively used in commercial rechargeable lithium-ion cells. This salt represents a compromise, but it is the only one that can largely meet the required property profile for technical use. The critical property of LiPF6 is that it is in chemical equilibrium with lithium fluoride (LiF) and phosphorus pentafluoride (PF₅), and the latter compound is extremely reactive and hygroscopic and will react with the traces of water, always present in the electrolyte in the range of <20 ppm, to form phosphoryl fluoride (POF₃) and hydrofluoric acid (HF). In very simplified terms, the POF₃ acts as a catalyst in the autocatalytic decomposition reaction of the carbonate-based solvents, the hydrofluoric acid contributes to the passivation of the positive current collector and the LiF precipitates as a solid due to its poor solubility and is also found in the SEI. The position of this reaction equilibrium depends strongly on the operational temperature and the residual moisture content of the electrolyte.

			,
Salt	Tm/°C	Tb/°C	Decomposition /Ageing
Lithium hexa fluoro phosphate [LiPF ₆]	200	80 (EC/DMC)	LiF, PF ₃ , PF ₅ , POF ₃ , HF

Table 5: SoA Li-salt for Lithium-Ion-Battery electrolytes

Table 5 lists the possible decomposition products of LiPF₆. Regarding the chemical reactivity of the active materials, it should be noted that they show different chemical reactivities depending on the state of charge, which can be well represented in a potential scale related to the normal hydrogen electrode (NHE) electrode. At a state of charge of 100%, the negative electrode shows a potential of ~-2.9V vs. NHE and the positive electrode a potential of 1.3V vs. NHE, which indicates that the former is an extremely strong reducing agent and the latter a strong oxidizing agent. For this reason, direct contact of oxidation- or reduction-sensitive components of the multi-sensor platform with the chemically highly reactive active material components, which are dependent on the charge state, should be avoided. These can be polymer materials, which are used for the purpose of insulating the sensor support lines, but also the sensor materials such as the e.g. glass-fiber material itself. On contact with e.g. a charged negative electrode, by a redox reaction, the glass fiber material would be chemically corroded on its surface and the negative electrode would be discharged to the same extent. The consequence would be on the one hand the loss of mobile lithium in the cell and on the other hand, which is probably more serious, the chemical and possibly structural change of the sensing area (probe) and thus the influence of the sensor signal. If the detection principle of the sensor itself is based on a chemical reaction, this would be massively disturbed by an additional chemically highly reactive reaction partner. It is therefore essential to take precautions for the careful separation of active materials and sensors.

It is self-evident that in the case of direct contact between the two electrodes, e.g. short circuit, the discharge reaction will occur thermodynamically voluntarily.

This brief description of the chemical environment in a lithium ion cell reveals that special precautions and a selection of suitable materials must be made for this environment. Analogous requirements must be met for the selection of suitable sensor materials. Here, too, it makes sense to distinguish between the actual sensor (sensor area), the sensor environment and the supply line. For all three sensor components, chemical resistance to electrolyte components and their ageing products must be ensured. This applies above all to the polymers and glass materials used. The second point concerns the possible contamination of the sensor area through local precipitation, deposition or cavity formation by gaseous decomposition products, which can lead to falsification of the sensor signal.

Sensor-ID	Sensor type	Sensor / Probe	Support	Supply Line	Disturbing/ Interfering	Consequences
				Material		
1	Fiber Bragg Grating (FBG)	Silica Fiber	Silica	Silica	Hydrofluoric acid (HF) → Silica Fiber Solid decomposition products → Sensing area Gaseous decomposition products → Sensing area	 fiber corrosion swelling/shrinkage embrittlement membrane plugging gas voids signal shift
2	Reference electrode	Li-metal, Li₄Ti₅O₁₂, LiFePO₄,	polymer non-woven, Cu/Al expanded Metal	Cu/Al expanded Metal	Organic solvent →Li- metal, Polymers	 electrode fouling (ageing) "electrolyte starved" cell signal shift
3	Luminescence	Silica Fiber	Polymers	Polymers	Hydrofluoric acid (HF) → Silica Fiber Organic solvent → Polymers Solid decomposition products → Sensing area Gaseous decomposition products → Sensing area	 fiber corrosion swelling/shrinkage embrittlement membrane plugging gas voids signal shift
4	Photo- acoustic	Infrared source (MEMS)	1	Cu/Al expanded Metal	Gaseous decomposition products → sensing area If the gas has infrared emission spectra of 4.2µm	- Noise in the signal

Table 6: Material selection for multi-sensor platform, issues, and consequences due to chemical environment

Table 6 gives an overview of the challenges and consequences for the selection of suitable materials for the sensor components. The chemical environment in a lithium-ion cell is subject to constant change, which must be considered in the design of the sensor systems.

Exclusion Criteria!

- ! Poor chemical stability against the chemical environment (chemical corrosion) in the cell which leads to total failure of the sensor and/or uncontrollable changes in the sensor capabilities.
- ! Contamination of the sensing area during operation by ageing products due to random local changes in the chemical environment.
- ! Direct contact between sensor line and highly chemically reactive active material (electrochemical redox reaction).
- ! No possibility to calibrate the multi-sensor platform.

2.1.5 Material dependent mechanical stress

Mainly responsible for the mechanical stress during the cycling of a lithium ion-cell are changes in the volume of the active materials depending on the state of charge. The main contributor to this volume change comes from the negative electrode. This volume changes can be transferred into a mechanical force for a given structural volume of the cells. The housing of the cells counteracts this force, however, especially in the case of prismatic cells (hard-case) as well as pouch cells (soft-case), an additional counterforce has to be applied to counteract the mechanical deformation of the cells. This so-called axial preload-force can usually be several kN/cell (e.g. 2N/mm²). For the multi-sensor platform, this has the consequence that the expected mechanical stress must be taken into account if the dedicated sensors are not used for force measurement.

Table 7 shows the percentage volume changes between SOC 0% and SOC 100% of the anode materials targeted to be used in the INSTABAT project.

Material	Graphite	L i4Ti5O12	Si
matorial	Orapinto		5
Volume change / %	10	0.2	270

Table 7: Volumes change of selected anode materials

If we recall Table 4, Table 5, it is easy to see that the anode and cathode are separated only by a relatively thin and porous polymer membrane that serves as an electron-separating ion conductor. In view of the expected linear expansion in the z-direction of the anode, the probability of the occurrence of high-impedance short circuits immediately increases, which are responsible for a high self-discharge rate and low Coulomb efficiency of the cell. Further prerequisite for this are additional process-related fluctuations during material and cell production. Nevertheless, for industrially manufactured cells with a SoA cell chemistry, which is used in the INSTABAT project, the relevant reject rate should be below 0.01%. The insertion of sensor tips including measuring leads between the anode and cathode, as outlined in Table 5, raises additional questions to those already discussed above. What dimensions may the sensor have, what is the permissible bias force, is additional separation necessary and how high will the failure rates be? Table 8 shows the expected and estimated failure rates due to high-impedance short circuits for the INSTABAT prototype cells depending on the technical maturity level. The failure rates can already be determined after activation by measuring the resistance and during the formation procedure of the cells by means of a short-circuit test.

 Table 8: Expected/estimated effect of strain, axial force on industrial pouch-cell, INSTABAT prototype cell, multi-sensor platform integrated into INSTABAT prototype cell

Platform/ Prototype-Cell*** Pouch-Cell*

	C/NMC622	C/NMC622	C/Si550++/NMC622	C/NMC622	C/Si550**/NMC622
Strain	high-impedance shorts (≤0.01%)	high-impedance shorts (≤5.0%)	high-impedance shorts (≤5.0%)	a. high-impedance shorts (≤10.0%) b. sensor failure (≤10.0%)	a. high-impedance shorts (≤10.0%) b. sensor failure (≤10.0%)
Axial Force	high-impedance shorts (≤0.01%)	high-impedance shorts (≤10.0%)	high-impedance shorts (≤10.0%)	a. high-impedance shorts (≤30.0%) b. sensor failure (≤30.0%)	a. high-impedance shorts (≤30.0%) b. sensor failure (≤30.0%)

*TRL9, ** TRL5, *** TRL4, **advanced (next generation) active material

In addition to the most probable risk, that of short circuits and fine circuits, massive mechanical damage in the form of cracks or complete breakage of the electrode foils and the sensors is also conceivable and must be assessed experimentally.

Exclusion Criteria!

- ! High-impedance short circuits for INSTABAT multi-sensor platform greater than 30%.
- ! Total rupture of the current collector or electrodes.
- ! The integration of the Multi-Sensor platform increases the safety hazard level to > 4.8

2.1.6 Temperature and current density distribution

For Li-ion cells under certain current loads, the format (pouch, cylindrical or prismatic) and construction-related current density distribution is of particular importance with regard to the thermal behavior (temperature-induced degradation) and the spatial electrochemical potential distribution of the electrodes (lithium-plating). Heat can dissipate from some locations more easily than from others. The poor through-plane conductivity of certain battery materials e.g. polymer-based separators causes higher heat accumulation in the core compared to the regions closer to the surface. Furthermore, the current densities and the heat generation rates are not always equal at different locations and strongly dependent on format and size. For cylindrical cells (e.g. 21700, 26650), the temperature in the cell core is significantly higher than at the surface. For pouch- or prismatic cells, the temperature is higher in the regions close to the tabs due to the higher current densities in those locations. The temperature near the positive tab is often higher than near the negative tab due to the higher ohmic resistivity of the aluminum current collector on the cathode side compared to copper on the anode side. The heat generation rate among the different regions is not uniform due to the non-uniformly distributed current during cycling. In course of mutual interaction an inhomogeneous current density can be caused by a temperature gradient and the location of tabs. High current density near the tabs may lead to local overcharge or over-discharge, potentially resulting in localized failure. The inhomogeneities in the temperature and current distribution can also lead to different local rates of side reactions and therefore different local degradation rates. Local temperature hot spots may cause high local temperature gradients with dramatically increased local current densities, leading to dendrite growth and eventual short-circuiting during charging. In Li-ion cells with graphite anodes charged at low temperatures the relationship between hot spots and dendrite growth rates may be more complex due to the interaction between temperature and the local potential of the graphite anode. In addition, the operating temperature has a significant influence on the phenomena discussed above. The premature local cell-aging caused by this and the undesired deposition of metallic lithium are significantly influenced by the choice of materials, the electrode processing and the cell design. These conditions must also be considered when selecting the sensors and their positions in the sensor platform. Ideally, the current density and temperature distribution can be

modeled over a wide C-rate and temperature range in order to be able to mode the measured values from the sensor signals for the complete cell.

Table 9 gives an overview of the necessities and limitations of sensor integration with regards to the real temperature and current density distribution to be expected. For each sensor type, a qualitative estimate of the dependence of the sensor signal to be measured on the anisotropy and the respective current density and temperature distribution in the cell is given.

Sensor-ID	Measured parameters	<i>Spatial-</i> environment	<i>Current</i> <i>density</i> - distribution	<i>Temperature</i> - distribution	Supporting model	Recommended measuring points
		anisotropy	depei	ndency	reco	mmended
	Temperature	high	high	high	yes	>1
1	Heat Flow	high	medium	high	yes	>1
	Pressure	low	medium	high	no	>1
	Strain	low	high	low	no	>1
2	"Absolute" potential	high	high	medium	yes	>1
2	Impedance	high	high	high	yes	>1
3	Temperature	high	high	high	yes	>1
J	Li+-concentration	medium	high	low	no	>1
4	CO ₂ -concentration	medium	low	medium	no	>1
5	E-based → Li ⁺ - concentration distribution, "absolute" potential	1	1	1	1	1
6	T-based → temperature, heat flow, pressure, strain	1	1	1	1	1

Table 9: Expected/estimated effect of current density and temperature distribution on capability of multi-sensor platform integrated into INSTABAT prototype cell

The selection of the optimal position for each of the sensors of the sensor platform must be made depending on the measured variable and the measuring principle. In this regard, an attempt should always be made to position the respective sensors outside of the stack or the roll. If such a positioning does not allow direct measurement of the desired measured variable, or a modeling with the data obtained is not valid enough, an attempt can be mat to go into the stack with the sensor tip. However, care must be taken to keep the loss of cell capabilities as minor as possible.

Excursus: Principle of ion concentration measurement

The principle of Li-ion sensor is detailed in Figure 9. A luminescent molecule able to catch the Li+ is used as a probe. The fluorescence of the molecule is not the same with and without Li+. These molecules are grafted on the surface of the optical fiber and can be excited by a certain range of wavelength (Excitation). The luminescence of the probe without Li ion is composed of multi peaks with different intensity. When the Li ion is complexed by the molecule, the presence of lithium absorbs some energy transition and reduces the associated intensity in the luminescence spectra. At the same time, the change in the molecule increases some of radiative transition. The change in

the spectra is proportional to the number of complexed probe and is directly related to the local concentration of Lithium ion near the probe.



Figure 9: Li-ion optical probe principle.

These recent reviews give more detail about the Li ion photoluminescent chemical probe and the mechanisms of detection^{9,10}. In the functionalized optical fibre (see Figure 9), the excitation light is coming from a source through the optical fibre to the end where the probes are chemically bounded. The probe is excited by evanescent wave created on the surface of the core without cladding. The luminescence emission of the probes is reinjected to the core by the same mechanism using evanescent wave. The intensity and the spectra of this collected luminescence depend on the number of complexed Li-ion on the probes.

In terms of environmental constraint, the optical fibre material and the probe should be chemically and thermally stable. The behaviour of the optical probe in terms of Li+ complexation/decomplexation should not be disturbed by the electrochemical cycle.

About the integration into the pouch cell, one of the major challenges is the point where the fibre goes through the sealing. The pouch is closed by heat-sealing after electrolyte feeling. Mechanical constraint and hot temperature during sealing should not affect the integrity of the fibre. The risk is to damage the cladding by thermal and mechanical degradation and also to fracture the silica core. This point needs to be addressed during the project. These constraints are the same for all the optical sensors based on fibres: FBG sensor, thermo-luminescent sensor and luminescent Li-ion sensor.

Exclusion Criteria!

- ! Multi-sensor platform allows only local information regarding a measurand in the INSTABAT prototype cell.
- ! No modelling of the measurand via the spatial extent of the INSTABAT prototype cell possible.
- ! Number of sensors required exceeds the effort for the benefit.

2.1.7 Advanced mechanical requirements for automotive application (optional beyond INSTABAT)

⁹ Villemin, E. & Raccurt O., Optical Lithium Sensors, Coordination Chemistry Review, 2021, accepted for publication
10 Kamenica, M.; Kothur, R.; Willows, A.; Patel, B. & Cragg, P., Lithium ion sensors, Sensors (Switzerland), 2017, 17

In addition to the mechanical requirements already explained in section 2.1.5, the material expansion caused by the cell chemistry, and the pre-tensioning force to be countered by this, further requirements due to the planned automotive application environment must be taken into consideration. Both for the cell alone and with the integrated sensor platform, its reliable functionality and safety must be ensured for such an environment. Two common tests for this are the vibration test and the acceleration test. From a safety point of view, these two tests provide answers to the question of leakage of the housing as well as the fire potential, e.g. due to provoked short circuits. For the multi-sensor platform, the question arises as to what effects high acceleration and application-related vibrations have on the positions of the sensor tips and consequently on their signals. Under no circumstances should a fire or explosion occur in the course of these tests. Due to the low technical maturity of the prototype cells and the multi-sensor platform/prototype cell, leakages as well as cell and sensor failures would be expected. The description of a possible test protocol can be found in chapter 8.

	Industrial- Pouch-Cell*	INSTABAT Prototype-Cell**		INSTABAT Multi-Sensor Platform/ Prototype-Cell***	
	C/NMC622	C/NMC622	C/Si550++/NMC622	C/NMC622	C/Si550++/NMC622
Vibration	a. no explosion/ fire b. no leakage c. cell failure (≤0.1%)	a. no explosion/ fire b. leakage (≤1.0%) c. cell failure (≤1.0%)	a. no explosion/ fire b. leakage (≤1.0%) c. cell failure (≤1.0%)	a. no explosion/ fire b. leakage (≤10.0%) c. cell/sensor failure (≤10.0%)	a. no explosion/ fire b. leakage (≤10.0%) c. cell/sensor failure (≤10.0%)
Shock	a. no explosion/ fire b. no leakage c. cell failure (≤0.1%)	a. no explosion/ fire b. no leakage c. cell failure (≤1.0%)	a. no explosion/ fire b. no leakage c. cell failure (≤1.0%)	a. no explosion/ fire b. leakage (≤10.0%) c. cell/sensor failure (≤20.0%)	a. no explosion/ fire b. leakage (≤10.0%) c. cell/sensor failure (≤20.0%)

 Table 10: Expected/estimated effect of vibration-test, and shock-test on industrial pouch-cell, INSTABAT prototype cell, multi-sensor platform integrated into INSTABAT prototype cell

*TRL9, ** TRL5, *** TRL4, **advanced (next generation) active material

For experimental details please refer section 8.

Exclusion Criteria!

- ! The integration of the Multi-Sensor platform increases the safety hazard level to > 4.8
- ! Total failure of the sensor and/or uncontrollable shift in the sensor capabilities

2.2 Electrical requirements

The electrical requirements for the INSTABAT multi-sensor platform/ prototype cell are defined on the basis of the technical maturity level (TRL). For approximate comparability, a hypothetical industrially manufactured pouch cell, corresponding to the a TRL9*, with the dimension and cell

chemistry of the INSTABAT prototype cell is used as the reference base. The predicted specifications for the INSTABAT prototype cell (TRL5^{**}) and the INSTABAT multi-sensor platform/ prototype cell (TRL4^{***}) are derive from this SoA-related configuration. According to the lower TRL, the quantitative performance indicators of the INSTABAT prototypes change compared to the hypothetical SoA specifications.

2.2.1 Capacity, Energy & Efficiency

The capacity and energy content are determined through a capacity test at **B**eginning **O**f **L**ife (BOL). For experimental details please refer section 5.3. Depending on the C-rate the discharge capacity must show at least the following values:

Table 11: Nominal capacity [Ah] for industrial pouch-cell, INSTABAT prototype cell, multi-sensor platform integrated into INSTABAT prototype cell

	Industrial- Pouch-Cell*	INSTABA ⁻	Γ Prototype-Cell**	INSTAB Platform/	AT Multi-Sensor Prototype-Cell***
	C/NMC622	C/NMC622	C/Si ₅₅₀ ++/NMC622	C/NMC622	C/Si ₅₅₀ ++/NMC622
0.5C	≥ 1.4	≥ 1.3	≥ 1.3	≥ 1.1	≥ 1.1
0.2C	≥ 1.4	≥ 1.4	≥ 1.4	≥ 1.3	≥ 1.3

*TRL9, ** TRL5, *** TRL4, **advanced (next generation) active material

Besides, the capacity dispersion within one cell batch must not exceed 2%*, 5%** and 10%***.

Exclusion Criteria ***TRL9!¹¹

! Capacity dispersion within one cell batch :

The measured energy density [Wh/dm³] and specific energy [Wh/kg] of the cells for 0.2C at BOL must be (see Table 12):

Table 12: Energy density [Wh/dm³] and specific energy [Wh/kg] for industrial pouch-cell, INSTABAT prototype cell, multi-sensor platform integrated into INSTABAT prototype cell

	Industrial- Pouch-Cell*	INSTABAT	Prototype-Cell**	INSTABAT Multi-Sensor Platform/ Prototype-Cell***			
	C/NMC622	C/NMC622	C/Si ₅₅₀ ++/NMC622	C/NMC622	C/Si550++/NMC622		
Wh/dm ³	500	475	525	450	500		
Wh/kg	220	210	220	200	210		

*TRL9, ** TRL5, *** TRL4, **advanced (next generation) active material

Besides, the energy dispersion within one cell batch must not exceed 2%*, 5%** and 10%***.

¹¹ *****TRL9** indicate a (hypothetical, beyond INSTABAT) industrial Multi-Sensor Platform/Cell and refer to the cell chemistry C/NMC622.

The energetic charge- and discharge efficiency between the defined voltage limits at 100% *Depth Of Discharge* (DOD) for *C/NMC622* cells must exceed the following values:

- **0.5C:** ≥ 95%*, ≥ 94%** and ≥ 92%***
- **0.2C:** ≥ 96%*, ≥ 95%** and ≥ 94%***

Exclusion Criteria ***TRL9! 11

- ! Reduction of specific energy and energy density >5%
- ! Energy dispersion within one cell batch >2%
- ! Energy efficiency <95% at 0.5C rate
- 2.2.2 Voltage range, nominal voltage and shipping state

The nominal voltage is determined at BOL. For experimental details please refer section *3.2.6*. For 0.2 C-rate discharge the nominal voltage must show at least the following values:

	Industrial- Pouch-Cell*	INSTABAT	Prototype-Cell**	INSTABAT Multi-Sensor Platform/ Prototype-Cell***				
	C/NMC622	C/NMC622	C/Si ₅₅₀ ++/NMC622	C/NMC622	C/Si ₅₅₀ ++/NMC622			
Operating voltage range	2.8 - 4.2V	2.8 - 4.2V	2.5 - 4.2V	2.8 - 4.2V	2.5 - 4.2V			
Nominal Voltage	3.6V	3.6 V	3.5 V	3.6 V	3.5 V			
Shipping state	30% SOC	30% SOC	30% SOC	30% SOC	30% SOC			

Table 13: Voltage range [V] nominal voltage [V] and shipping state [%SOC] for industrial pouch-cell, INSTABAT prototype cell, multi-sensor platform integrated into INSTABAT prototype cell

*TRL9, ** TRL5, *** TRL4, **advanced (next generation) active material

2.2.3 Internal resistance

For experimental details please refer *section 5.5.* The internal DC-resistance of the C/NMC622 cells at 50% SOC must be ≤ 12 mOhm^{*}, ≤ 22 mOhm^{**} and ≤ 30 mOhm^{***}.

In addition, the resistance dispersion of all cells within one cell batch must not exceed 2%*, 10%** and 20%***.

Exclusion Criteria ***TRL9! 11

- ! Increase DC-resistance at *BoL* >5%
- ! Resistance dispersion within one cell batch >2%

2.2.4 Currents

The maximum continuous currents for charging and for discharging (linked with internal resistance, please refer section 2.2.3).

Table 14: Maximum continuous currents [A] for industrial pouch-cell, INSTABAT prototype cell, multi-sensor platform integrated into INSTABAT prototype cell

	Industrial- Pouch-Cell*	INSTABAT	Prototype-Cell**	INSTABAT Multi-Sensor Platform/ Prototype-Cell***		
	C/NMC622	C/NMC622	C/Si ₅₅₀ ++/NMC622	C/NMC622	C/Si ₅₅₀ ++/NMC622	
Charge	≥ 4.2 A (3C)	≥ 2.8 A (2C)	≥ 2.8 A (2C)	≥ 1.4 A (1C)	≥ 1.4 A (1C)	
Discharge	≥ 7.0 A (5C)	≥ 4.2 A (3C)	≥ 4.2 A (3C)	≥ 2.8 A (2C)	≥ 2.8 A (2C)	

*TRL9, ** TRL5, *** TRL4, **advanced (next generation) active material

Exclusion Criteria ***TRL9! 11

! Maximum current decreases (accepting loss of power) by ≥5% due to increased DC-resistance

2.3 Environmental requirements

Basically, in terms of environment requirement, a distinction can be made between the environment in the cell and the operational environment of the cell. The former concern all components and materials required for the functioning of the cell and the sensor, the latter address the cell a whole and include climatic conditions such as temperature and humidity. The temperature exerts the most significant influence udder the external conditions on the performance indicators and the aging of cells.

2.3.1 Operating temperature range

For all Cell-types *,**,*** the allowed temperature range during operation must be between **0°C** and **+60°C** for charging and between **-20°C** and **+60°C** for discharging.

2.3.2 Temperature at Storage

For all Cell-types ****** during storage, temperatures between -20 and +60°C are required.

Exclusion Criteria ***TRL9! 11

- ! Operative temperature range for charging would be limited to > 0°C and < +60°C
- ! Operative temperature range for discharging would be limited to > -20°C and < +60°C

2.4 *Lifetime requirements*

The lifetime requirements shall provide provisional forecast of calendrical and cyclic aging. Ultimately, the lifetime depends on the materials selection, manufacturing quality, operating environment, and the power profile.

2.4.1 Calendar Life

The calendrical aging determination is made after one month of storage at 100% SOC and two different temperatures. For experimental details please refer section *6.1*. Here, the following irreversible capacity loss must not be exceeded:

- 23°C: ≤ 1,0%*, ≤ 2,0%** and ≤ 3,0%***
- 60° C: ≤ 3,0%*, ≤ 5,0%** and ≤ 7,0%***

The self-discharge after one month must not exceed the following values:

- 23°C: ≤ 2,0%*, ≤ 3,0%** and ≤ 5,0%***
- 60° C: ≤ 6,0%*, ≤ 8,0%** and ≤ 10,0%***

Exclusion Criteria ***TRL9! ¹¹

! Calendrical aging \geq 1,0% month⁻¹ at 23°C and \geq 3,0% month⁻¹ at 60°C ! Self-discharge \geq 2,0% month⁻¹ at 23°C and \geq 6,0% month⁻¹ at 60°C

2.4.2 Cycle Life

For experimental details please refer section 6.2.

integrated into INSTABAT prototype cell								
	Industrial- Pouch-	INSTABAT Prototype-Cell**	INSTABAT Multi-Sensor Platform/ Prototype-Cell***					

Table 15: Expected cycle life for industrial pouch-cell, INSTABAT prototype cell, multi-sensor platform

	Pouch- Cell*	INSTABAT	Prototype-Cell**	INSTABAT Multi-Sensor Platform/ Prototype-Cell***		
	C/NMC622	C/NMC622	C/Si ₅₅₀ ++/NMC622	C/NMC622	C/Si ₅₅₀ ++/NMC622	
Cycles	≥1500	≥ 700	≥ 300	≥ 400	≥ 200	
% DOD	90%	90%	90%	90%	90%	
Temperature	25°C	25°C	25°C	25°C	25°C	
C-Rate	0.5 / 0.5	0.5 / 0.5	0.5 / 0.5	0.5 / 0.5	0.5 / 0.5	

*TRL9, ** TRL5, *** TRL4, **advanced (next generation) active material

Here the End Of Life (EOL)-criterion is $\leq 80\%$ Q_{BOL} or a doubling of the direct current internal resistance (DC-IR).

Exclusion Criteria ***TRL9! 11

Cycle life (90%DOD, 25°C) ≤ 1500; Q_{EOL}= 80% SOH, DC-IR = 200% DC-IR_{BOL}

2.5 Cell safety

The cell (only applicable for cylindrical and prismatic hard-case cells) can have an internal, from the **b**attery **m**anagement **s**ystem (BMS) independent, safety element but it is not a requirement. The choice depends on the outcome of the safety tests. For **INSTABAT** all Cell-types *,**,*** must pass the following safety tests analogous to the accompanying test specification with a **Hazard Level** \leq **4** (according to ^[12]). For each experiment at least three cells must be tested.

2.5.1 Short-circuit test

For experimental details please refer section 7.1.

The cell must be short-circuited with a resistance of $\leq 1 m \Omega$

2.5.2 Overcharging

For experimental details please refer section 7.2.

The overcharging test is analogous to [12] with 1C overcharge current to 200% SOC or twice the maximum charge voltage (2 x V_{max}).

2.5.3 Nail penetration

For experimental details please refer section 7.3.

The nail test is performed with a 3.0 mm steel nail and a nail speed of 80 mm/s.

2.5.4 Thermal stability test

For experimental details please refer section 7.4.

At the 120 °C plateau the cell must exhibit a consistent Hazard Level of \leq 3.

At the 150 °C plateau the cell must exhibit a consistent Hazard Level of ≤ 4 .

The informative test result at the 200 °C plateau is taken out of any review and assessment.

Exclusion Criteria ***TRL9! ¹¹

! Sensor integration would increase the Safety Hazard Level > 4.8

3 General test specification

3.1 General for cells

¹² Sandia Report SAND2005-3123 – FreedomCAR: Electrical Energy Storage System; Abuse Test Manual for Electric and Hybrid Electric Vehicle Applications

This specification describes the test conditions necessary for the characterization of INSTABAT Prototype-Cells** and INSTABAT Multi-Sensor Platform/ Prototype-Cells***. The general test specifications for the multi-sensor platform are provisional and may change in course of the ongoing project in accordance with the latest findings in consultation with the project partners.

The applicable test procedures and sequences are described in more detail here. The specified test conditions relate to a test environment temperature of 25 ± 2 °C and to BOL conditions, unless otherwise specified.

Electrical, mechanical & safety tests	INSTABAT Prototype-Cell**	INSTABAT Multi-Sensor Platform/ Prototype-Cell***
Production		
Formation / Capacity test	(x)	(x)
Maturing / End classification	(x)	(x)
Basic Characterization		
Cyclic aging at 0,5C/0,5C; 25°C; 10%SOC-90%SOC	(0)	(0)
Cyclic aging at 1C/1C; 25ºC; 10%SOC-90%SOC	(0)	(0)
Cyclic aging at 0,5C/0,5C; 25ºC; 100% DOD	(x)	(x)
Cyclic aging at 1C/1C; 25ºC; 100% DOD	(0)	(0)
Capacity test (depending on C-rate)	(x)	(x)
HT - Storage (60ºC, 30d)	(x)	(X)
Advanced characterization (beyond INSTABAT	Project)	
Capacity test (depending on temperature and C-rate)	(0)	(0)
Mechanical requirements (shock, vibration)	(0)	(0)
Safety		
Overcharging	(x)	(x)
Nail penetration	(0)	(0)
Thermal stability test	(x)	(x)
Short-circuit test	(0)	(0)
(x) Scope of INSTABAT Project		
(o) Option (beyond INSTABAT Project)		

Table 16: Test overview for INSTABAT prototype cell and multi-sensor platform integrated into INSTABAT
prototype cell

3.2 **Definitions**

3.2.1 Nominal capacity

The nominal capacity (Q_{nom}) is the medium 0.2C discharge capacity (100% DOD) at 25°C specified by the cell manufacturer.

3.2.2 BOL capacity

The "Begin of Life" capacity (Q_{BOL}) is either the one specified by the cell manufacturer or it is determined through a standard cycle at the beginning of a test series. The capacity will be defined through a complete 0.2C discharge (100% DOD) in the 4th cycle at 25°C. See also 5.2.

3.2.3 Actual capacity

The actual capacity (Q_{actual}) is always the current capacity of an (aged) cell during/after a test series.

3.2.4 Current rates

Current rates are stated in relation to the nominal capacity. Discharge currents are declared with a negative sign (-), charge currents with a positive sign (+).

3.2.5 Energy

The energy ($E_{100\% DOD}$) is the medium 0.2C discharge energy (100% DOD) at 25°C specified by the cell manufacturer.

3.2.6 Nominal voltage

The nominal voltage($V_{nominal}$) results at 50% SOC and 0.2C discharge in the 4th cycle. It is calculated as follows:

$$V_{nominal} = \frac{E_{100\% DOD}}{Q_{50\% SOC}}$$

3.2.7 Open circuit voltage

The open circuit voltage (OCV) is measured at no load and after a relaxation time of 30 min.

3.2.8 Shipping state

The shipping state of the cell is 30% SOC.

3.2.9 SOH und EOL-criterion

The "State Of Health" SOH describes the aging state of a cell and is determined by:

$$SOH = \frac{Q_{actual}}{Q_{BOL}} * 100\%$$

The "End Of Life" EOL criterion corresponds to the SOH depicted in the Product Requirement Document (see 2.4.2).

e.g. $Q_{actual} \le 80\% Q_{BOL}$ (SOH $\le 80\%$) and/or DC-IR_{actual} $\ge 200\%$ DC-IR_{Bol}

4 Geometric dimensions and tolerances

The cells dimensions must be measured with a calibrated measuring device.

The dimensions of the cell must be within the specified measurement and tolerance values.

5 Electrical tests

5.1 Standards

5.1.1 Charging

The CC/CV charging is performed with the defined current to the maximum charging voltage and a CV-cut-off current of C/20.

5.1.2 Discharging

The CC discharging is performed with the defined current until the maximum discharging voltage is reached.

5.1.3 Pause periods

The pause periods for capacity testing, SOC settings, etc. are 30 minutes between each half cycle. The pause periods serve for relaxation and cooling purposes.

5.2 **Cycle**

A cycle consists of a charging and subsequent discharging process along with defined dead times/pause periods.

5.3 Capacity test

A capacity test for determining the actual capacity comprises 4 charge / discharge cycles. After three initial cycles the capacity is determined in the 4th discharge cycle. In addition to the capacity the energy efficiency is determined by considering the charging and discharging energy:

$$\eta = \frac{E_{Discharge}}{E_{Charge}} * 100\%$$

Remark:

In special cases, this capacity test can also be performed in the 4^{th} cycle with a 0.2C in order to determine the corresponding capacity and energy efficiency. The results for 1C are obtained in the 3^{rd} cycle.

5.4 Setting the SOC

The setting of a desired SOC takes always place after a capacity test and a charging procedure. Subsequently, the desired amount of charge has to be extracted from the cell (or added), followed by a 30 min relaxation pause. Additional SOCs can be approached without renewed capacity check.

5.5 Nominal DC internal resistance test (R_{DC})

The DC internal resistance shall be determined at an SOC of 50% and 25°C. Prior to the test the cell shall be stored for at least one hour. The cell shall be discharged at a constant current I_1 of 0.2 C. After 30 seconds the cell voltage U_1 under load shall be recorded. Subsequently the discharge current shall be immediately increased to a value I_2 of 1.0 C. The corresponding cell voltage under load U_2 shall be measured after 1 and 10 seconds.

The DC internal resistance is calculated as follows

$$R_{DC} = \frac{U_1 - U_2}{I_2 - I_1}$$

5.6 Capacities at different temperatures

Before a temperature-dependent test the cell must be in a thermal equilibrium. The equilibrium is reached when for the duration of one hour, the difference between the measured cell temperature and the test environmental temperature does not exceed 2 °C.

For each of the required temperatures a test with 4 cycles and a subsequent internal resistance check are carried out.

The sequence for one cell begins and ends with a 25 °C capacity test, in order to obtain BOL / EOL reference values and to gain conclusions on the aging.

Further measurements run from warm to cold temperatures.

Example for a temperature sequence: $25^{\circ}C \rightarrow 40^{\circ}C \rightarrow 10^{\circ}C \rightarrow 0^{\circ}C \rightarrow -10^{\circ}C \rightarrow 25^{\circ}C$

The temperature-dependent capacities are divided by the BOL-capacity in order to get the relative ratio. The following formula illustrates the determination at 0°C:

$$\eta_{0^{\circ}C} = \frac{Q_{0^{\circ}C}}{Q_{BOL}} * 100\%$$

Analogous to this procedure the functioning at other operating temperatures (e.g. -20 ° C) can be checked.

5.7 Capacity test at various continuous C-rates

For each of the required C-rates a capacity test with 4 cycles is carried out. The C-rate for charging and discharging are examined asymmetrically. Unless otherwise defined the following rates are applied: 0,2C; 0,5C; 1C; 2C

The C-rate-dependent capacities are divided by BOL-capacity in order to get the relative ration. The following formula illustrates the determination at 2C cycling:

$$\eta_{2C} = \frac{Q_{2C}}{Q_{BOL}} * 100\%$$

6 Aging tests

6.1 Calendaric aging

6.1.1 Pre-conditioning

After a capacity test (BOL) according to section 5.3 the cell is charged to 100% SOC.

6.1.2 Storage

At the required temperature (25°C; 60°C; ...) the cell is stored for at least 30 days.

6.1.3 Discharge process

After cooling to room temperature (RT) the cell is discharged with 0.2C in order to determine the self-discharge (see also 5.1.2). Then a new capacity and internal resistance check with 4 cycles takes place.

6.1.4 Evaluation

In order to evaluate the calendaric aging both capacities of the 4th cycle, the one before and after storage, are set in ratio in order to determine the SOH (see also 3.2.9) respectively the irreversible capacity loss.

irrev. capacity loss = 1 - SOH

The self-discharge is determined according to the ratio of the first discharge capacity after storage and the BOL capacity from the pre-conditioning.

capacity loss =
$$(1 - \frac{Q_{1\text{st Discharge after storage}}}{Q_{BOL}}) * 100\%$$

6.2 Cyclic aging

6.2.1 Pre-conditioning/reference values

In order to determine the cyclic aging, the capacity as well as the internal resistance at the beginning (BOL), at the end of the test series and in appropriate intermediate steps are measured. These reference values are crucial for determining the SOH values. If the EOL criterion is reached the cyclization can be cancelled.

6.2.2 Cycling

The cycling itself, takes place at the respective test temperature, the required C-rate for the demanded number of cycles. These values are provided in the product requirement document. The cell is charged and discharged in the full voltage range (100% SOC), unless otherwise defined.

7 Safety tests

Test protocols and specifications for the safety tests may adapted according to the actual requirements and findings during the INSTABAT project.

All in this chapter described safety tests are performed with fully charged cells (100% SOC). The number of cells per test is defined in the product requirement document (see 2.5).

The following parameters are recorded in their chronological sequence:

- Cell voltage
- Currents
- Temperatures of terminals; cell surface; clamping plates; surroundings
- Others: Nail temperatures; gas pressures etc.

Moreover, the critical testing process is documented by video recording. In tests with no visible event a photo is taken at the end of the test procedure in order to proof the fault-free state of the cell for documentation purposes.

7.1 Short-circuit test (optional beyond INSTABAT)

The short-circuit test is carried out on a fully charged cell (100% SOC). With a resistance $\leq 1 \text{ m}\Omega$, the cell is short-circuited and observed. After 10 minutes, the short circuit has to be released again and the cell has to be observed until it has cooled down sufficiently (≤ 50 ° C).

7.2 **Overcharging**

The overcharge test is carried out on a fully charged cell (100% SOC). The cell is overcharged with a current of 1C to 200% SOC. The overcharge current is maintained up to twice the charge voltage or until a security element triggers. Subsequently, the cell has to be observed for 1h.

If the test was successful (no thermal runaway), the cell is to be transferred to a safe state.

7.3 Nail penetration (optional beyond INSTABAT)

The nail penetration test is performed with a 3.0 mm steel nail which is centrally and perpendicular to the largest winding side inserted to the cell with a nail speed of 80mm / s to 1 mm before reemergence of the nail.

The test is terminated when the cell is cooled down again (\leq 50 ° C).

7.4 Thermal stability test

The thermal stability test is carried out in a temperature-testing furnace. Therein, the cell is preconditioned to 80 °C (for max. 16h).

The cell is heated and kept at a 120 °C plateau for 2h. Afterwards a 150 °C Plateau follows for also 2h. The test is then formally ended but can be restarted with a 200 °C Plateau for 30 min in order to gain further information.

The test is terminated when the cell is cooled down again (\leq 50 ° C).

8 Mechanical tests (optional beyond INSTABAT)

8.1 Vibration

The cell should be charged according to standard charged at 25°C \pm 2°C, and standby 30 minutes at 23°C \pm 2°C. Cell to be load surely to testing machine and vibrated for 90 minutes for each of the three mutually perpendicular planes with total excursion of 1.15mm and with frequency of 10 Hz to 55 Hz. Cell to be discharged, at 23 \pm 2°C with constant discharge current 0.2C until cut off voltage (e.g. 2.5V)

8.2 Shock

The cell is to be secured to the testing machine by means of a rigid mount which supports all mounting surfaces of the cell. Each cell shall be subjected to a total of three shock are to be applied in each of three mutually perpendicular directions shall be tested. Each shock is to be applied in a direction normal to the face of the cell. For each shock the cell is to be accelerated in such a manner that during the initial 3 milliseconds the minimum average acceleration is 75g (where g is the local acceleration due to gravity). The peak acceleration shall be between 125g and 175g. Cells shall be tested at a temperature of $25\pm2^{\circ}C$

9 Concluding remarks and outlook

A long service life combined with high practical benefits and economic affordability are the main key enabler for high acceptance of electrochemical energy storage solutions among OEMs as well as consumers. A long lifetime additionally contributes to the fulfillment of current environmental sustainability demands and increases consumer satisfaction. The need for convenience must be met with high functionality and performance. The often-underestimated key factor, a highly automatable production opens economic perspectives that enable many market participants to enter the market as well as economic access to this technology. The most essential of these demands, especially those related to technological aspects, could perhaps be met by the "smart cell" concept, which could be realized with the utilization of integrated sensor solutions, as an inevitable key technology. Key aspects as a prerequisite for this were addressed and discussed in the previous chapters, and limitations and exclusion criteria were elaborated. If the production of battery cells with integrated sensor technology can be automated to such an extent that economic concerns in terms of investment costs can be put aside, the potential benefits must be included in the analysis on an equal footing with the question of potential production costs. Essential research work to answer these subject areas is being carried out within the INSTABAT project in the task areas of WP6. There, both the techno-economic feasibility and the market potential for use cases will be investigated. But even before such further questions are answered, criteria can be defined or derived on the basis of the current state of knowledge that can be expected to pose a serious obstacle to further promising integration of sensor technologies in battery cells in an early project phase or in the case of low technical maturity.

To draw attention to these issues again, the following table provides a summary of the restrictions that must be prevented or circumvented. In this list, aspects concerning industrialization, scalability and transferability are left out. For this, reference is made to WP6, which starts with M24.

Table 1	17:	List	of	challenges	and	limitations	for	integrating	the mult	i-sensor-	platform	into cells

ID	Description	Impact
1	The integration of the Multi-Sensor platform increases the safety hazard level to > 4	Standards/RegulationsOEM and consumer acceptance
2	Reduction of the energy density and specific energy by > 15% through integration of the multi-sensor platform	
3	High-impedance short circuits and consequently high failure rate through integration of the multi-sensor platform	- OEM and consumer acceptance
4	Sensor with a larger cross-section in the z-direction than 2 times the electrode diameter	 Increase of mechanical stress Increase in cell-failure probability
5	Total rupture of the current collector or electrodes	- Increase in cell-failure probability
6	Safety-critical deposition of lithium at locations where the sensors are integrated into the stack	- Impact on safety hazard level
7	Poor chemical stability against the chemical environment (chemical corrosion)	- Failure of the sensor
8	Contamination of the sensing area during operation by ageing products due to random local changes in the chemical environment	 Onpredictable changes in the sensor capabilities OEM and consumer acceptance
9	Direct contact between sensor line and active material and highly chemically reactive active material (electro-chemical redox reaction)	 Impact on safety hazard level Increase in cell-failure probability Failure of the sensor
10	Gas and moisture tightness of the feedthroughs for the multi-sensor platform is not guaranteed	 Premature (cell/sensor)-aging Failure of cell and sensor
11	No possibility to (re)-calibrate the multi-sensor platform	
12	Multi-sensor platform allows only local information regarding a measurand	- OEM and consumer acceptance
13	No sufficiently accurate modelling of the measurand via the spatial extent of the cell possible	
14	Number of sensors required exceeds the effort for the benefit	