The following SOP for 2032 coin cell assembly and cycling (formation, aging test, rate capability test, self-discharge test, calendar aging test) was used in the BIG-MAP project. The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 957189).

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The images are also part of the PhD thesis of Matheus Leal de Souza, defended in January 2024

Standards and Protocols for the BIG-MAP project

Those protocols targeted 2032 coin cells, with the following chemistries:

Cathode: NMC811, LNO, LFP

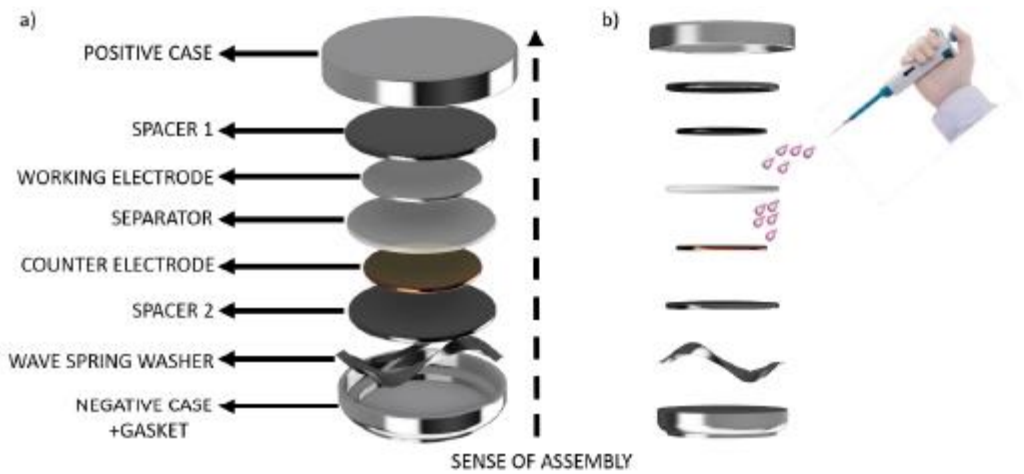
Anode: Gr, Si/Gr (11% : 74% of the electrode mass)

Coin cell assembly:

It follows the scheme below, for thin film separators (such as Celgard)

Volume of Electrolyte is ideally between 4 and 6 times the electrode minimal porosity. Minimal porosity is defined as (anode porous volume + cathode porous volume + separator porous volume). Electrolyte is added twice: first above the anode, and then over the separator for optimized spread.

1 stainless steel spacer is added between the case and the working electrode, and between the spring and the counter electrode. Its thicknesses are as such as they guarantee good contact between the stack and both cases when cell is sealed. The ideal stack height is 1.10 times (not less than 1.03 or more than 1.28) thicker than that of the empty cell (only positive and negative cases) height when sealed. For that, the thicknesses of each case are considered to calculate the stack height.



Basic cells cycling:

All of the protocols below were concepted as a general procedure, but they may be modified to attend specific electrode or electrolyte chemistries, and specific cycling conditions.

<table>
<tr><th>FORMATION CYCLES</th><th>CYCLES QUANTITY</th><th>CHARGE PHASE</th><th>DISCHARGE PHASE</th><th>DATA RECORD</th></tr>
<tr><td>Full-cell and Positive half-cell</td><td rowspan="2">3 Cycles</td><td>CC C/10;
CV $i_{cutoff} < C/50$;
5 min OCV</td><td>CC D/10;
5 min OCV</td><td>5 mV and each 30s</td></tr>
<tr><td>Negative half-cell</td><td>CC D/10;
CV $i_{cutoff} < D/50$;
5 min OCV</td><td>CC C/10;
5 min OCV</td><td>5 mV and each 30s</td></tr>
</table>

Table 1 Formation cycles protocol for full-cell, positive half-cell or negative half-cell.

The cycling protocol below is just an example of the targeted cycling conditions focused on the BIG-MAP project experimental study. The charge or discharge phase currents could be modified to evaluate cell degradation under the intended current rate of study.

<table>
<tr><th>AGING FROM CYCLING</th><th>FORMATION CYCLES</th><th>CYCLES QUANTITY</th><th>CHARGE PHASE</th><th>DISCHARGE PHASE</th></tr>
<tr><td>Full-cell and Positive half-cell</td><td rowspan="2">Follows table 1</td><td rowspan="2">80% SOH is reached at least</td><td>CC 1C;
CV $i_{cutoff} <$ C/20;
5 min OCV</td><td>CC 1D;
5 min OCV</td></tr>
<tr><td>Negative half-cell</td><td>CC 1D;
CV $i_{cutoff} <$ D/20;
5 min OCV</td><td>CC 1C;
5 min OCV</td></tr>
</table>

Table 3 Aging from cycling protocol for full-cell, positive half-cell or negative half-cell.

The rate tests are asymmetric, meaning that there is one D-rate (same charge and different discharge currents), and one C-rate test, that could be operated independently.

<table>
<tr><th>RATE TEST</th><th>FORMATION CYCLES</th><th>CYCLES QUANTITY</th><th>CHARGE PHASE</th><th>DISCHARGE PHASE</th></tr>
<tr><td rowspan="8">D-rate</td><td rowspan="16">Follows table 1</td><td rowspan="16">5 cycles for each varying current density</td><td rowspan="8">CC C/20 (or CC C/10; $i_{cutoff} < C/20$);
5 min OCV</td><td>CC D/20;</td></tr>
<tr><td>CC D/10;</td></tr>
<tr><td>CC D/5;</td></tr>
<tr><td>CC D/2;</td></tr>
<tr><td>CC 1D;</td></tr>
<tr><td>CC 2D;</td></tr>
<tr><td>CC 5D;</td></tr>
<tr><td>CC D/20;</td></tr>
<tr><td rowspan="8">C-rate</td><td>CC C/20;</td><td rowspan="8">CC D/20;
5 min OCV</td></tr>
<tr><td>CC C/10;</td></tr>
<tr><td>CC C/5;</td></tr>
<tr><td>CC C/2;</td></tr>
<tr><td>CC 1C;</td></tr>
<tr><td>CC 2C;</td></tr>
<tr><td>CC 5C;</td></tr>
<tr><td>CC C/20;</td></tr>
</table>

Table 4 Rate capability initial protocols for D-rate and for C-rate. During the varying current density cycles, a 5 min OCV is applied in the end of the phase of charge or discharge.

RATE TEST	FORMATION CYCLES	CYCLES QUANTITY	CHARGE PHASE	DISCHARGE PHASE
D-rate	Follows table 1	4 cycles for each varying current density	CC C/10; i_{cutoff} < C/20); 5 min OCV	CC D/10;
				CC D/5;
				CC D/2;
				CC 1D;
				CC 2D;
				CC 5D;
				CC D/10;
C-rate			CC C/10;	CC D/10; 5 min OCV
			CC C/5;	
			CC C/2;	
			CC 1C;	
			CC 2C;	
			CC 5C;	
			CC C/10;	

Table 5 Rate capability final protocols for D-rate and for C-rate. During the varying current density cycles, a 5 min OCV is applied in the end of the phase of charge or discharge.

	FORMATION CYCLES	AGING FROM CYCLING STEP	CHARGE PHASE	DISCHARGE PHASE
SELF-DISCHARGE	Follows table 1	20 Cycles following table 3	CC 1C; CV i_{cutoff} < C/20; 1h OCV	CC 1D; 5 min OCV
			CC 1C; CV i_{cutoff} < C/20; 10h OCV	CC 1D; 5 min OCV
			CC 1C; CV i_{cutoff} < C/20; 20h OCV	CC 1D; 5 min OCV
			CC 1C; CV i_{cutoff} < C/20; 40h OCV	CC 1D; 5 min OCV
			CC 1C; CV i_{cutoff} < C/20; 160h OCV	CC 1D; 5 min OCV and loop to aging from cycling step

Table 6 Self-discharge protocol for a cell in OCV at 100% SOC at the end of the CCCV charge step.

For the calendar aging, a time length of 1 week was employed, but this could be enlarged for periods of 1 month, for example.

	FORMATION CYCLES	REFERENCE CYCLE	CHARGE PHASE	DISCHARGE PHASE
CALENDAR AGING (SHELF-LIFE)	Follows table 1	1 Cycle CC C/10; CV $i_{cutoff} < C/20$; 5 min OCV CC D/10; CV $i_{cutoff} < D/20$; 5 min OCV	CC C/10; CV $i_{cutoff} < C/20$; 168h (1 week) OCV	CC D/10; CV $i_{cutoff} < D/20$; 5 min OCV Loop to charge phase until end of criteria

Table 7 Calendar aging protocol for a cell in OCV at 100% SOC.