

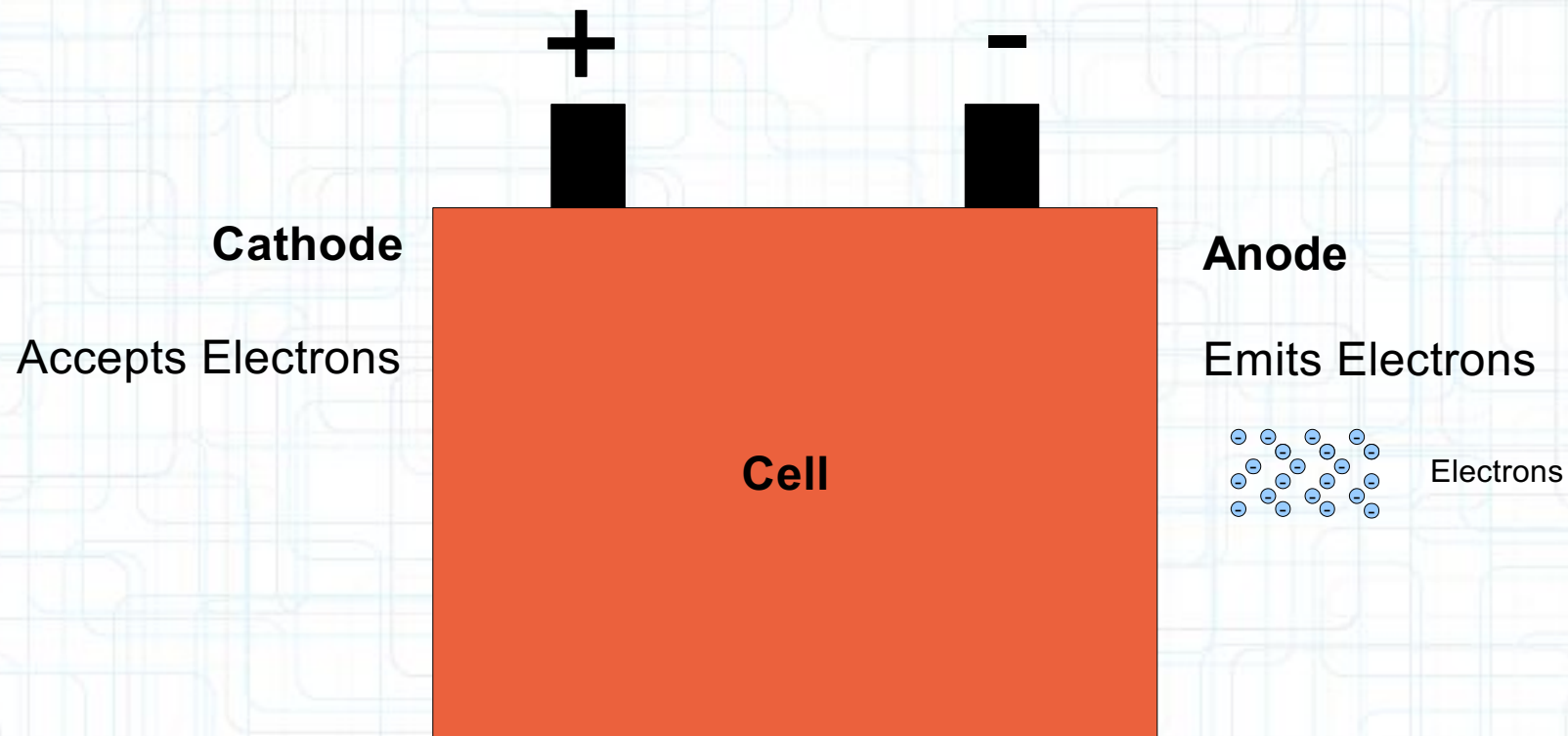
Lithium Polymer Battery Technology

Introduction

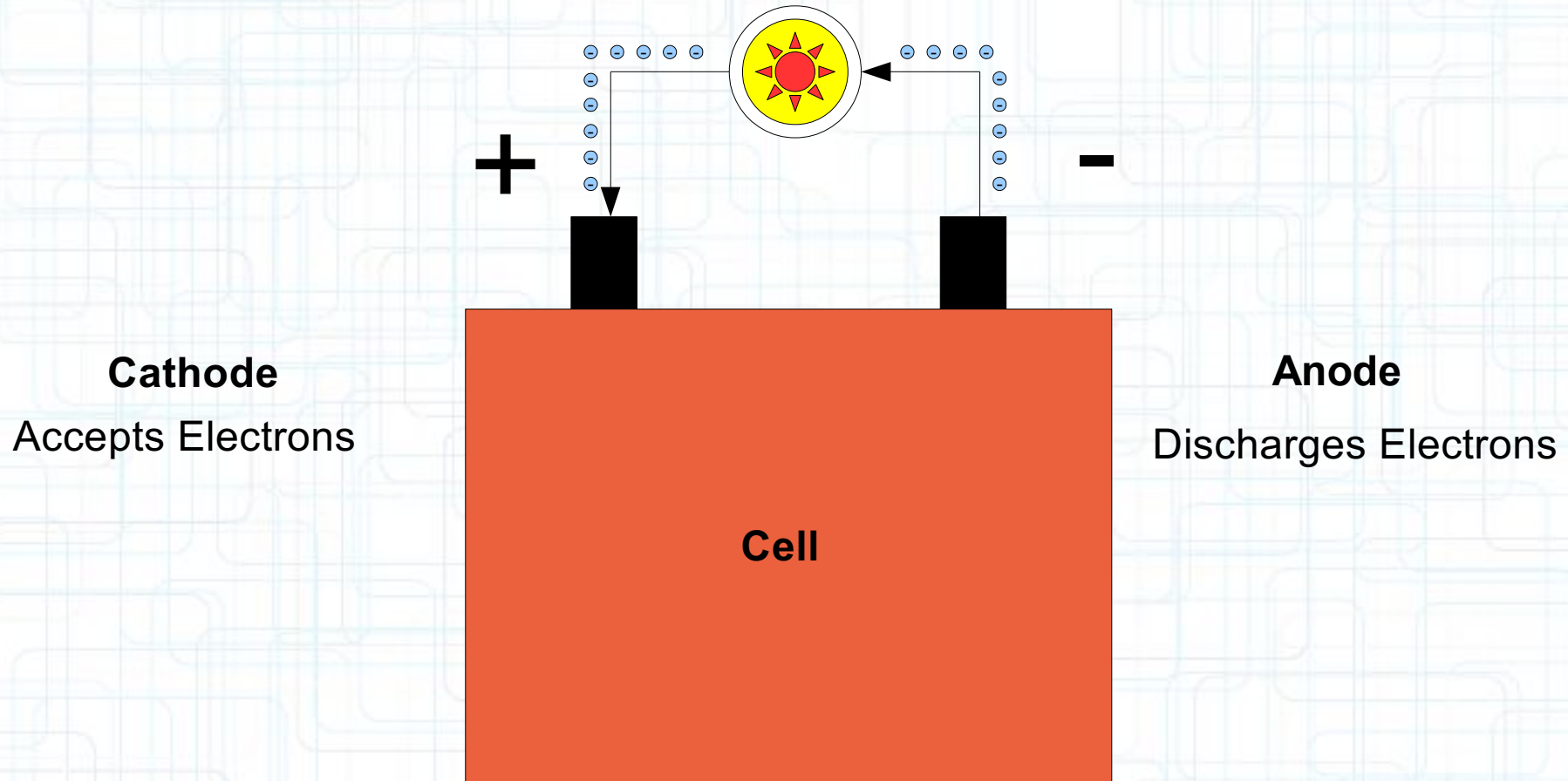
Dipl.-Ing. Frank Siegert

Transcribed by Julian Luyt

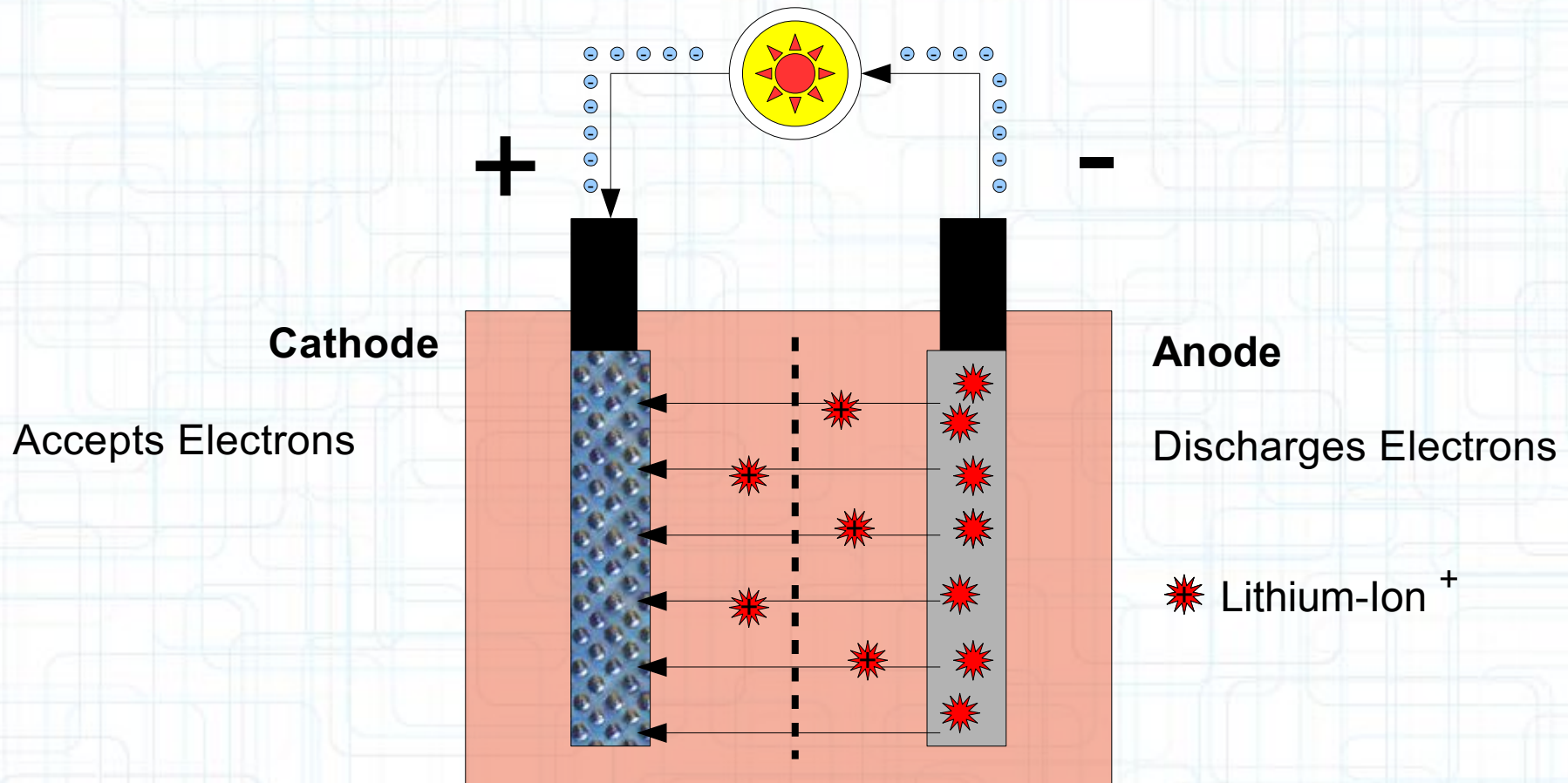
Overview



Discharge Cycle



Discharge cycle electron migration



Electrons flow outside
 Li^+ ions migrate within the electrolyte

For electrons, the cell is a non-conductor

Which particles migrate in the cell?

The Periodic Table of the Elements

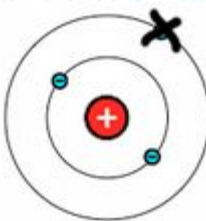
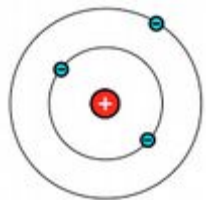
Legend:

- alkali metals
- alkaline metals
- other metals
- transition metals
- lanthanoids
- actinoids
- metalloids
- nonmetals
- halogens
- noble gases
- unknown elements
- radioactive elements have masses in parenthesis

Notes:

- as of yet, elements 113-118 have no official name designated by the IUPAC.
- 1 kJ/mol = 96.485 eV
- all elements are implied to have an oxidation state of zero.

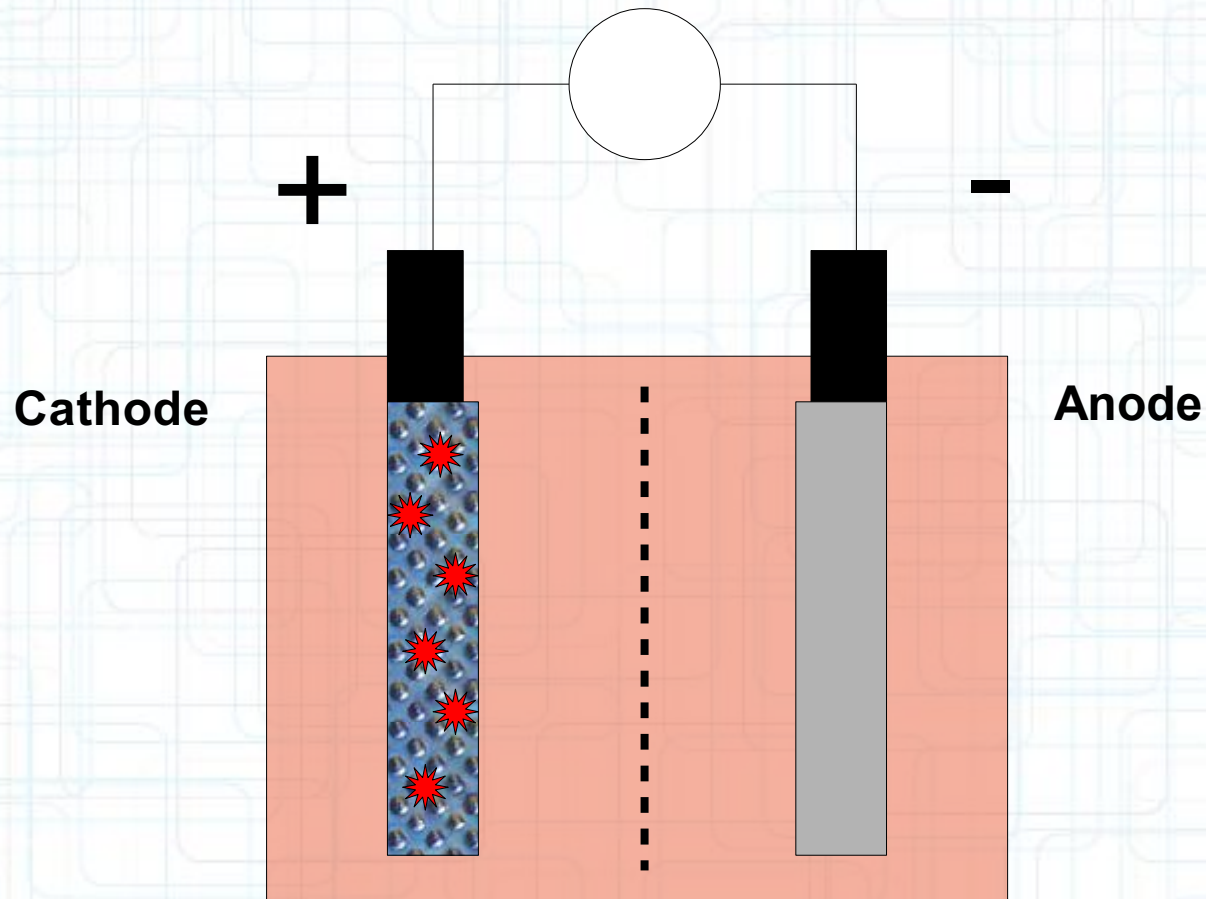
From Wikimedia Commons, the free media repository



Lithium to hydrogen and helium, the lightest element ,
An alkali metal, 3 protons in the nucleus, 3 electrons in the shell

Lithium-ion has 3 protons in the nucleus but only 2 electrons
in the shell. Making it positively charged.

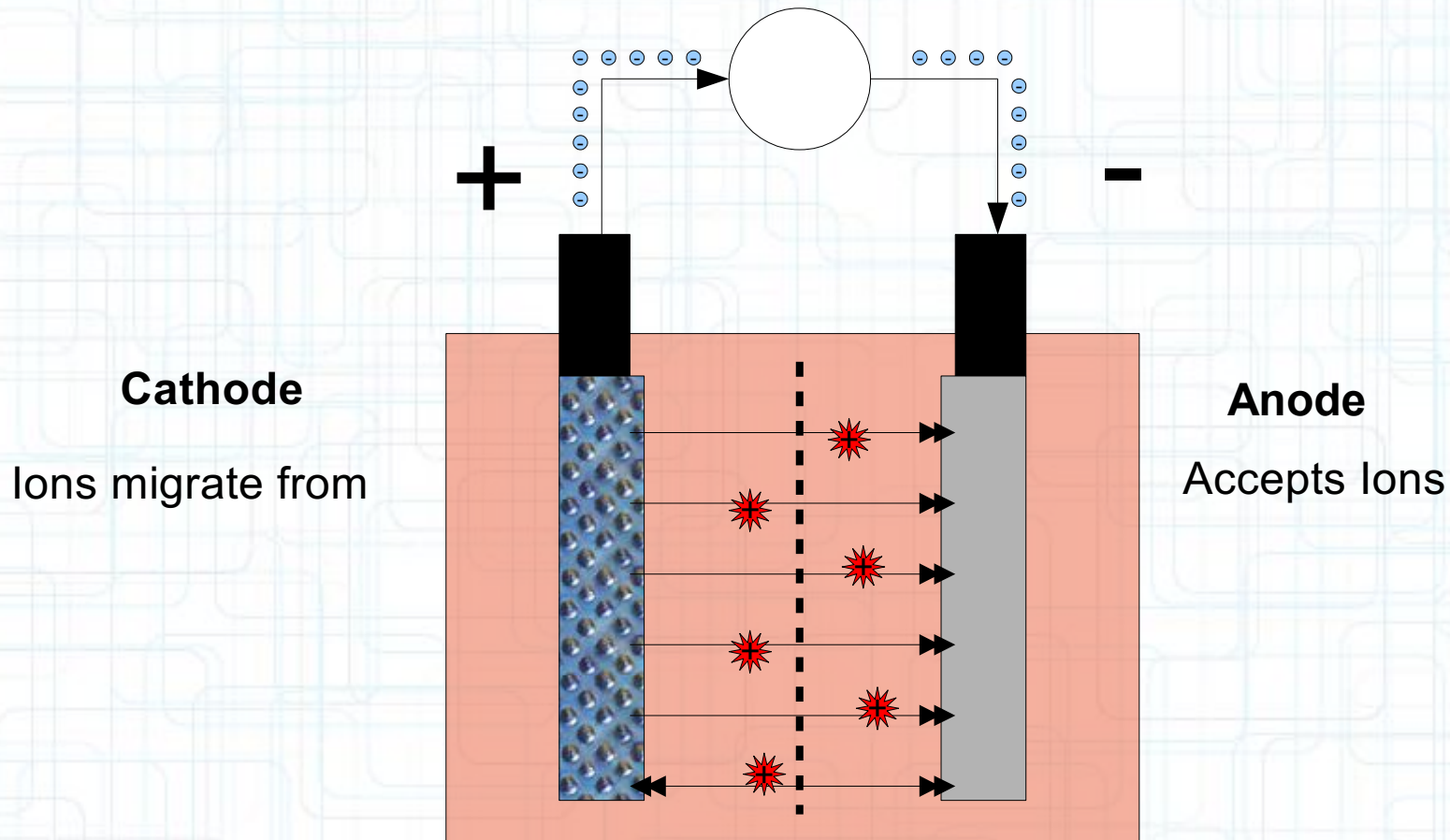
A totally discharged cell



The cell strives to reach the state of "lowest energy".

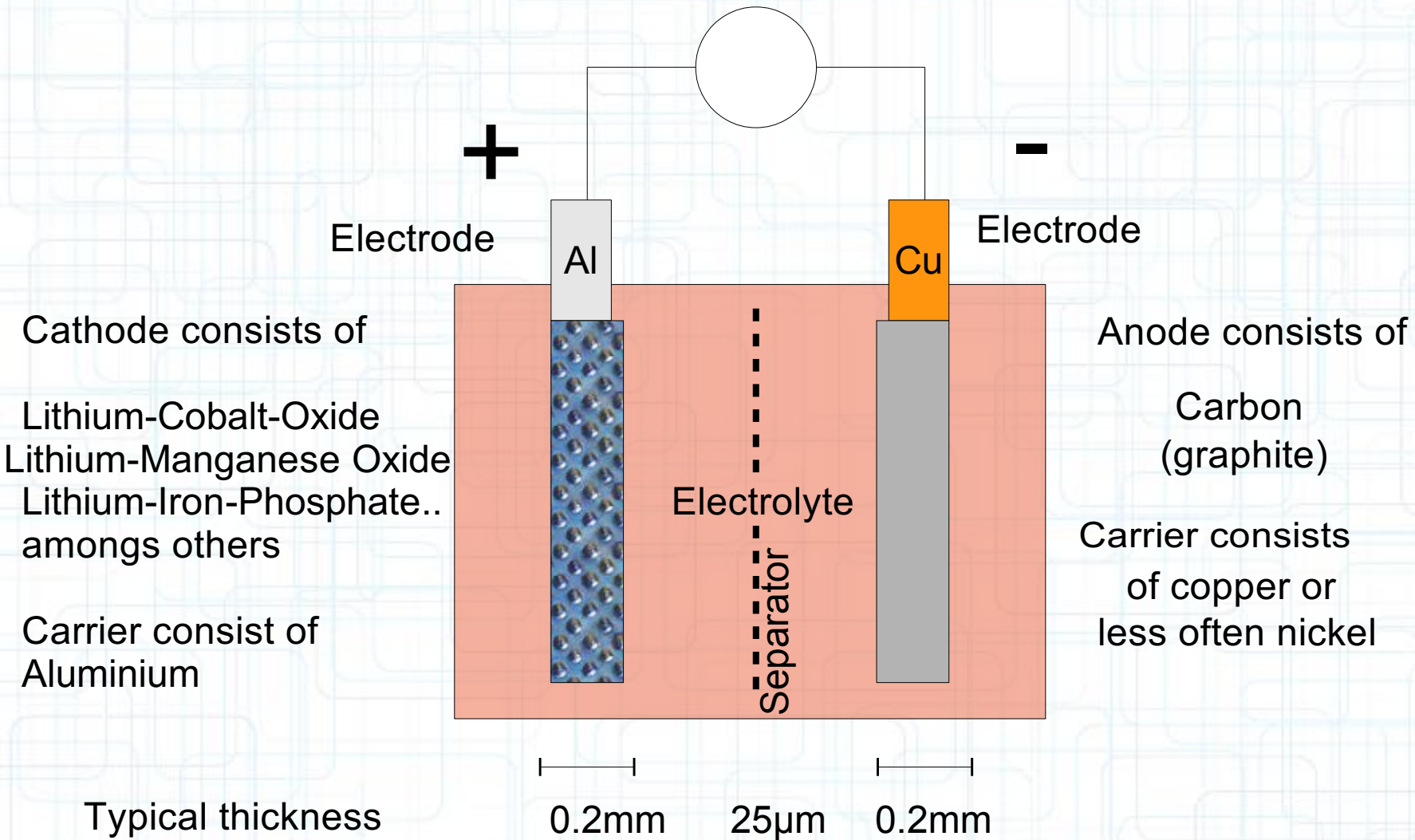
This is (theoretically) achieved when the cathode is replenished with lithium

Charging Cycle



When charging electrons flow to the anode,
 Li^+ ions migrate to the anode

Cell Structure



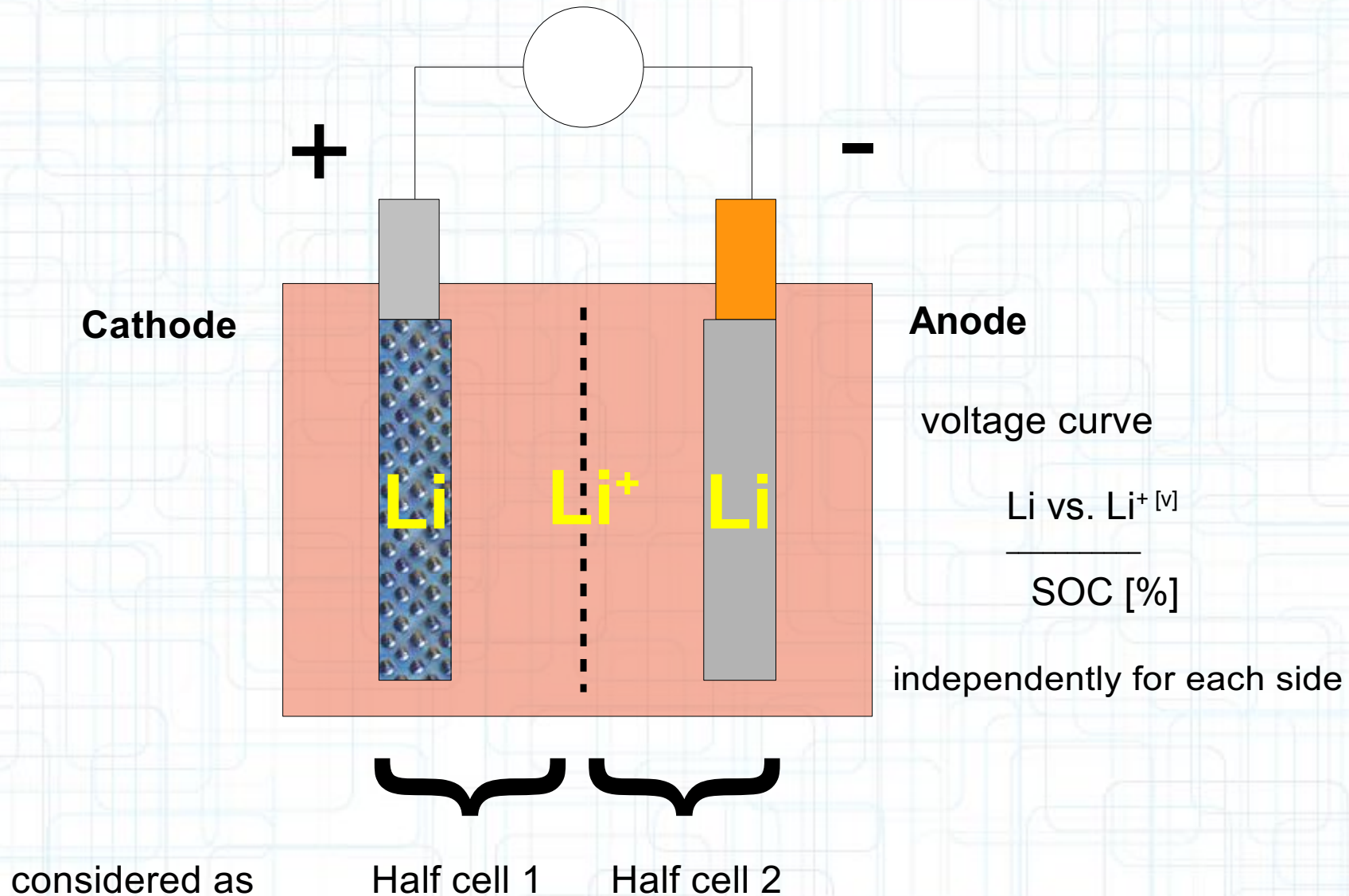
Cell structure

At a one amp current flow per second, the ion migration rate is:

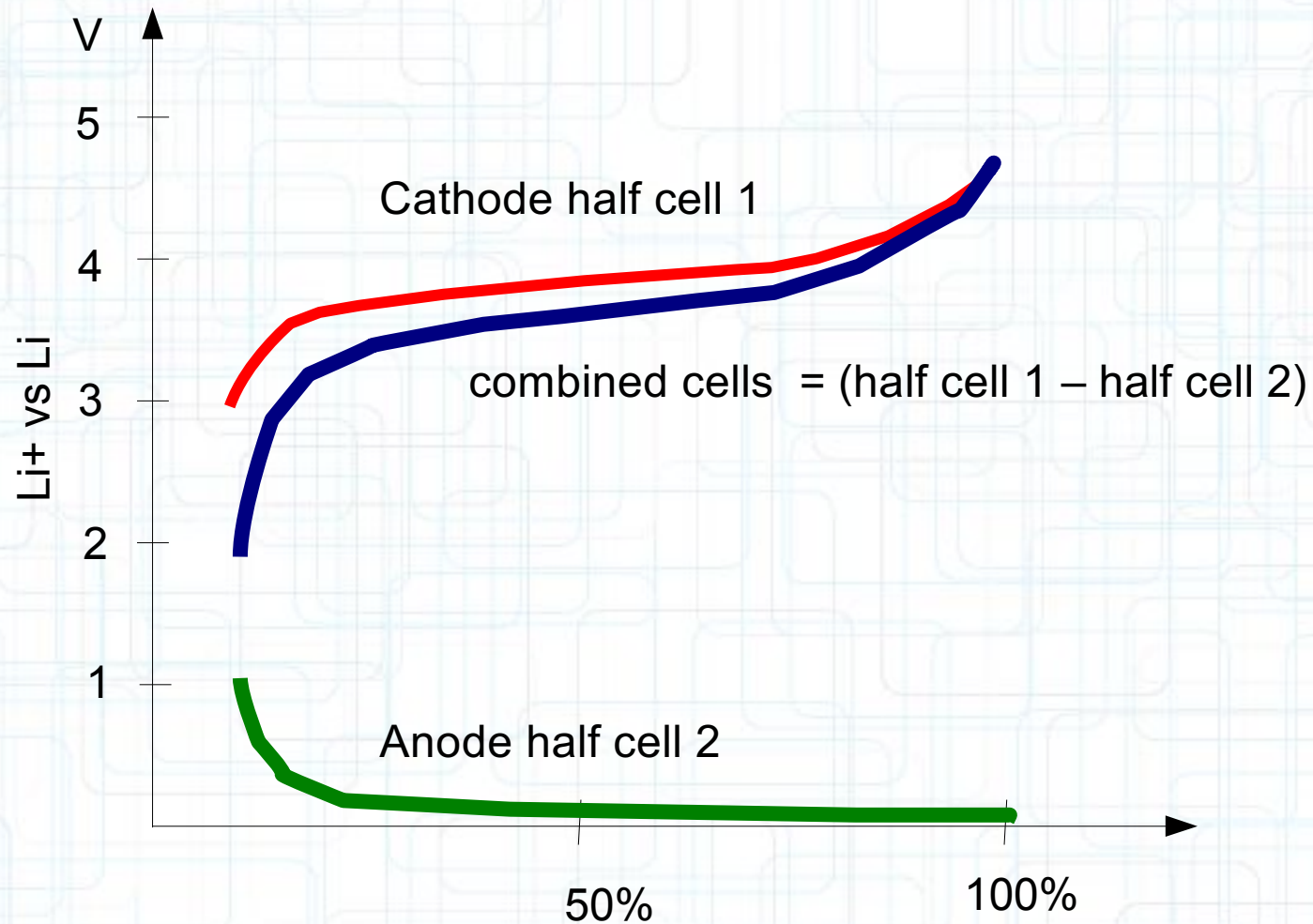
$$6.2 * 10^{18}$$

Li⁺ ions through the cell

Voltage Curve

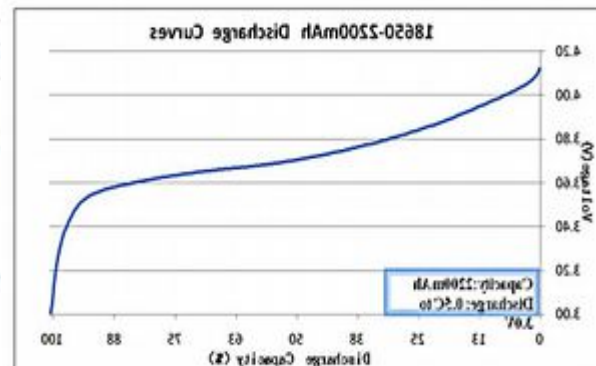
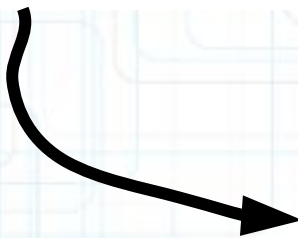
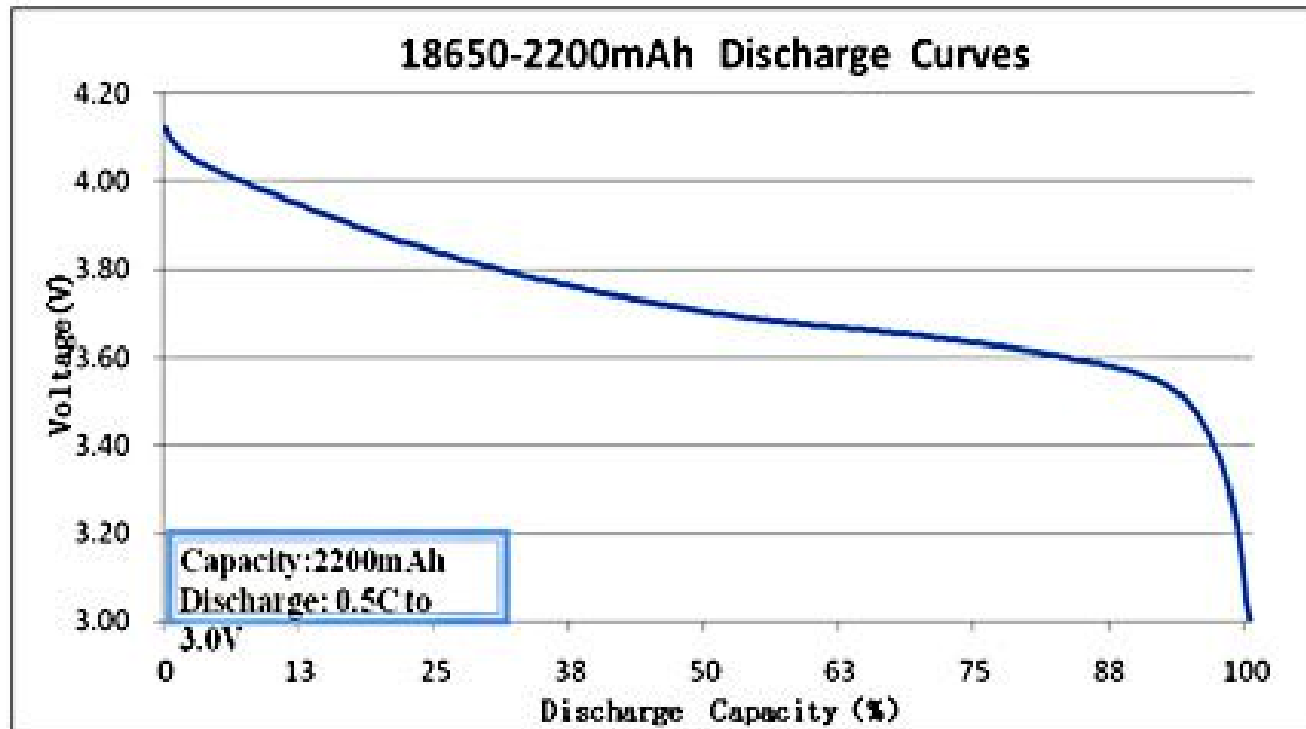


Voltage Curve Theory



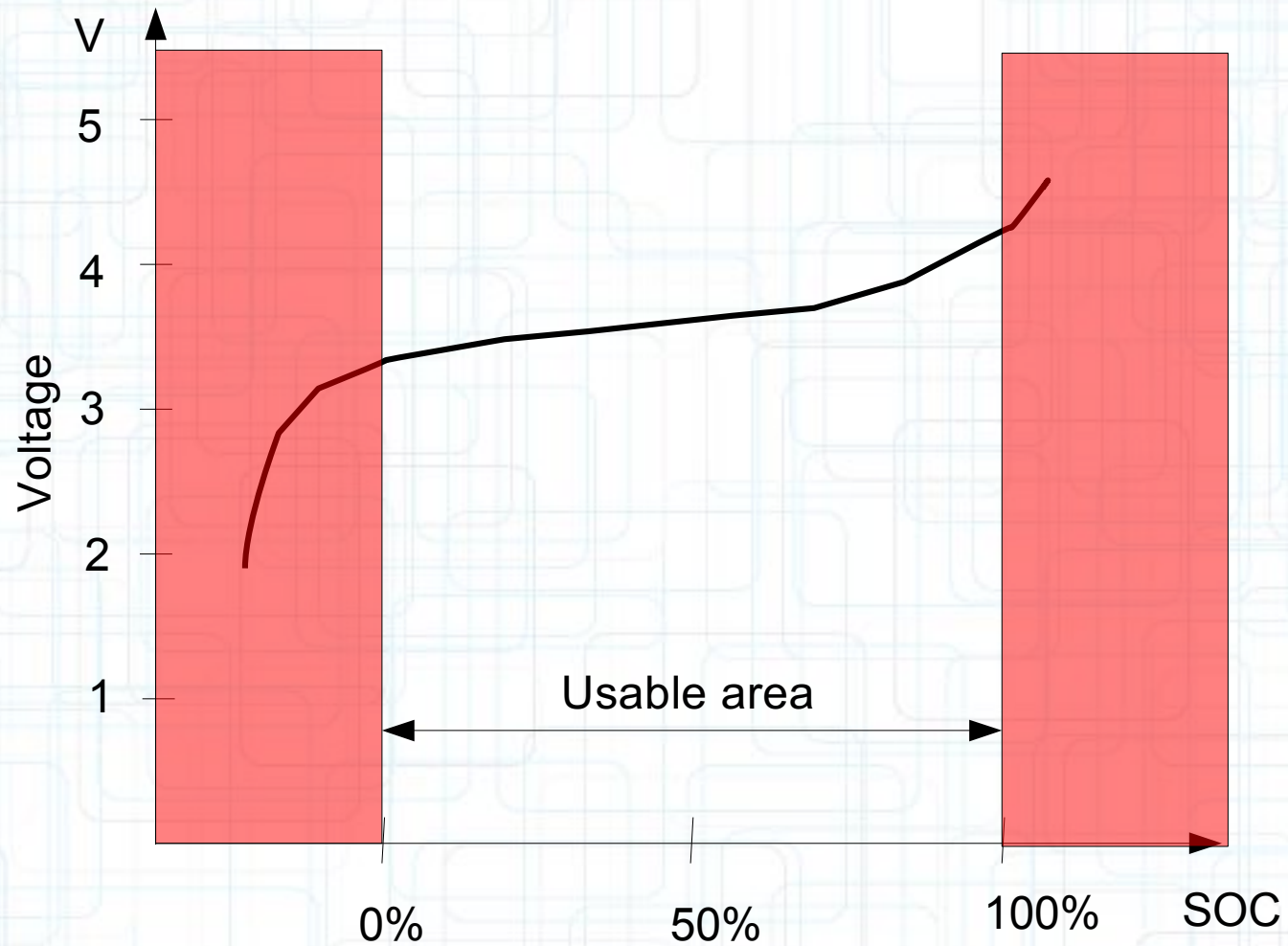
Voltage curve measured

Voltage in the unloaded state is a measure of the state of charge



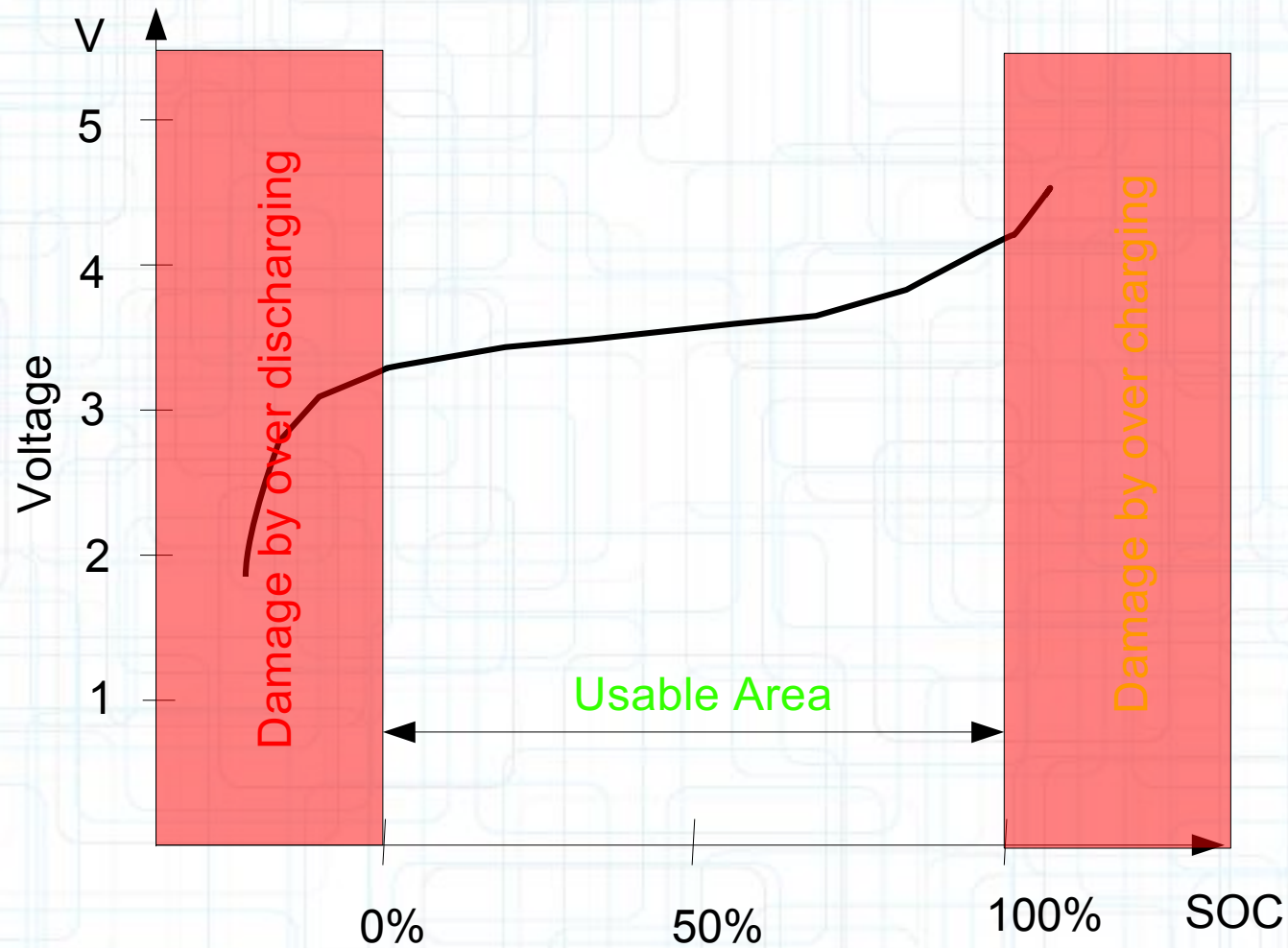
Mirror image of discharge graph trace to SOC graph trace

Voltage Curve SOC



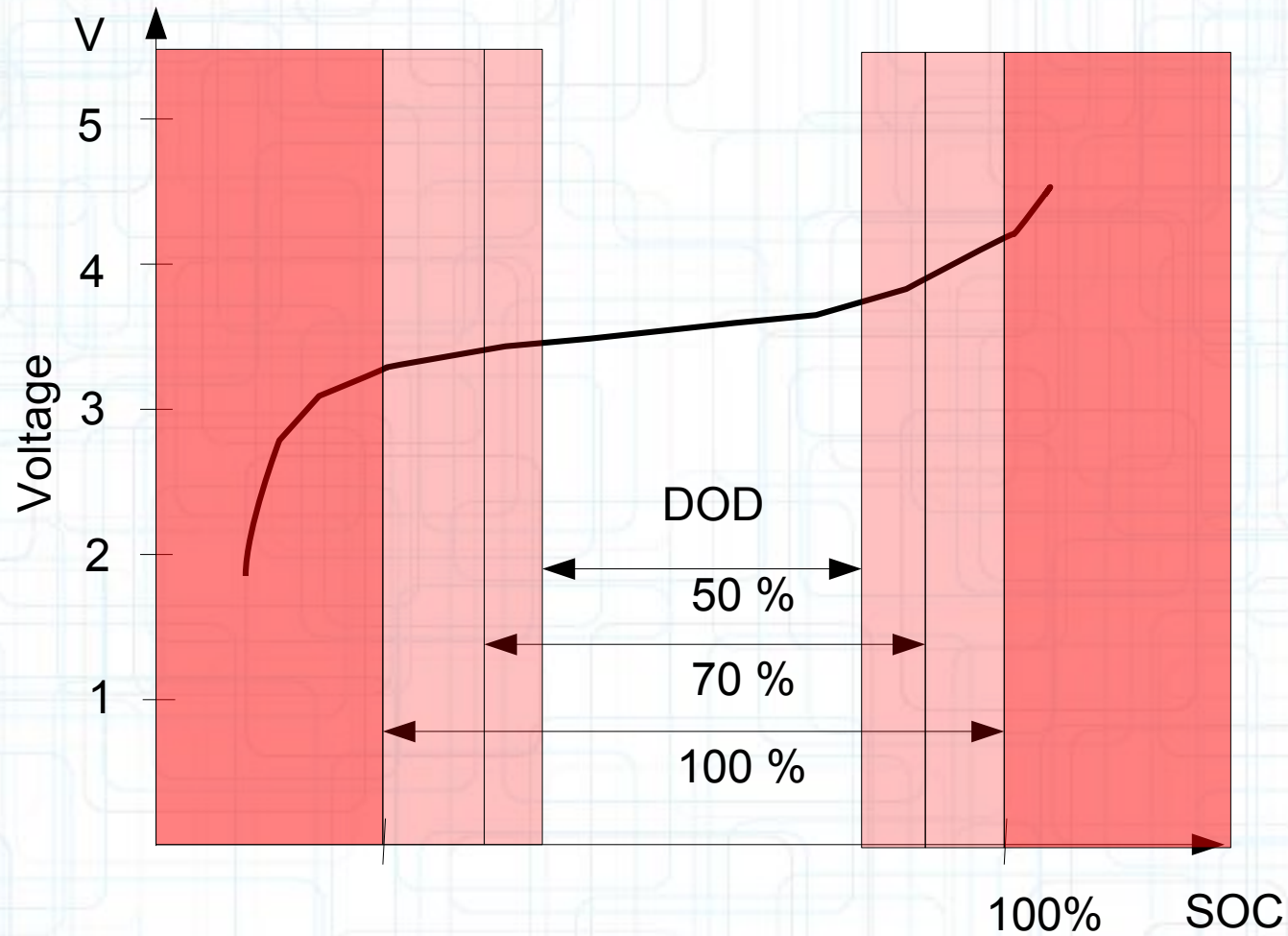
SOC = State of Charge

Voltage Curve SOC



SOC = State of Charge

Voltage Curve DOD



DOD = Depth of Discharge
Utilization rate of max. capacity available

Aging Cycles

Depth of discharge	Discharge cycles	Table 2: Cycle life as a function of depth of discharge A partial discharge reduces stress and prolongs battery life. Elevated temperature and high currents also affect cycle life.
100% DoD	300 – 500	
50% DoD	1,200 – 1,500	
25% DoD	2,000 – 2,500	
10% DoD	3,750 – 4,700	

Quelle: Batteryuniversity.com

Maximum utilization → Minimum lifetime

Above figures for ~ 1C discharging.

High-current discharges lead to a greatly reduced cycle life by accumulation of damage in the cell.

Maximum current drain → Minimum lifetime

Aging during storage periods

Temperature	40% charge	100% charge	Table 3: Estimated recoverable capacity when storing Li-ion for one year at various temperatures Elevated temperature hastens permanent capacity loss. Not all Li-ion systems behave the same.
0°C	98%	94%	
25°C	96%	80%	
40°C	85%	65%	
60°C	75%	60% (after 3 months)	

Quelle: Batteryuniversity.com

Deterioration due to aging depends on

Storage voltage (higher than 3.8V/cell is faster)

Storage temperature (higher the faster it ages)

Aging

The total life depends on:

Cycle aging

Damage in the cell by charging/discharging

Storage aging

Damage in the cell due to aging (storage)

$$\text{Aging}^{\text{Total}} = \text{aging}^{\text{Cycle}} + \text{aging}^{\text{storage}}$$

Aging

The effect of aging:

capacity decreases

Internal cell resistance increases

Current delivery capability decreases

Maximum possible charging current decreases

Aging occurs non linearly, after a certain degree of "cell damage", the deterioration occurs more rapidly (see section "Cell Chemistry" for details)

Mechanical construction

- Pouch Cell
- Layered or wrapped
- In a plastic pouch
- Filled with electrolyte



Quelle: Wikipedia

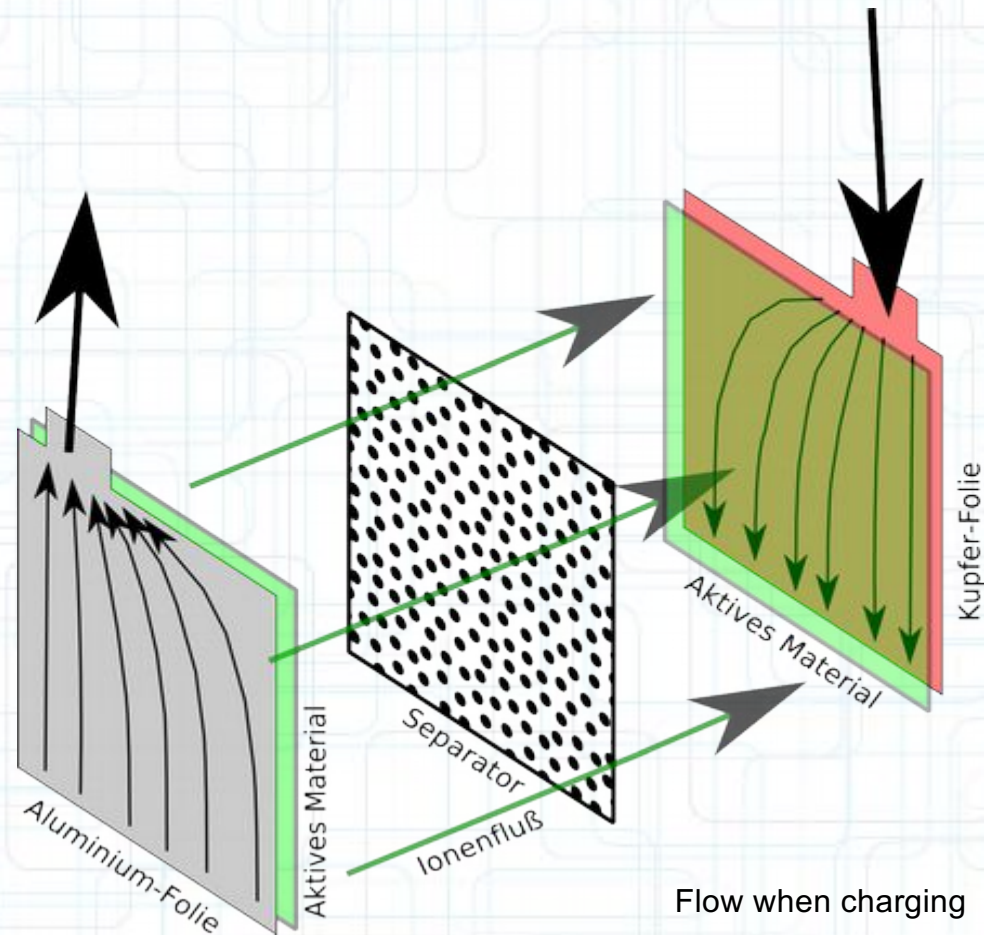
Physical construction

Thin layers, wrapped, folded or 'stacked' for maximum surface area



Quelle: Wikipedia

Current / Ion migration



The current flow in the cell is not equally distributed, but concentrates

near the terminals:

Grey Terminal-Aluminium Foil

Copper Red Terminal-Copper Foil

Heat development

The heat development of the cell during charging and discharge is composed of two elements:

Irreversible Heat

Due to the ohmic resistance, and electrochemical processes during overcharging/over discharging.

Reversible Heat

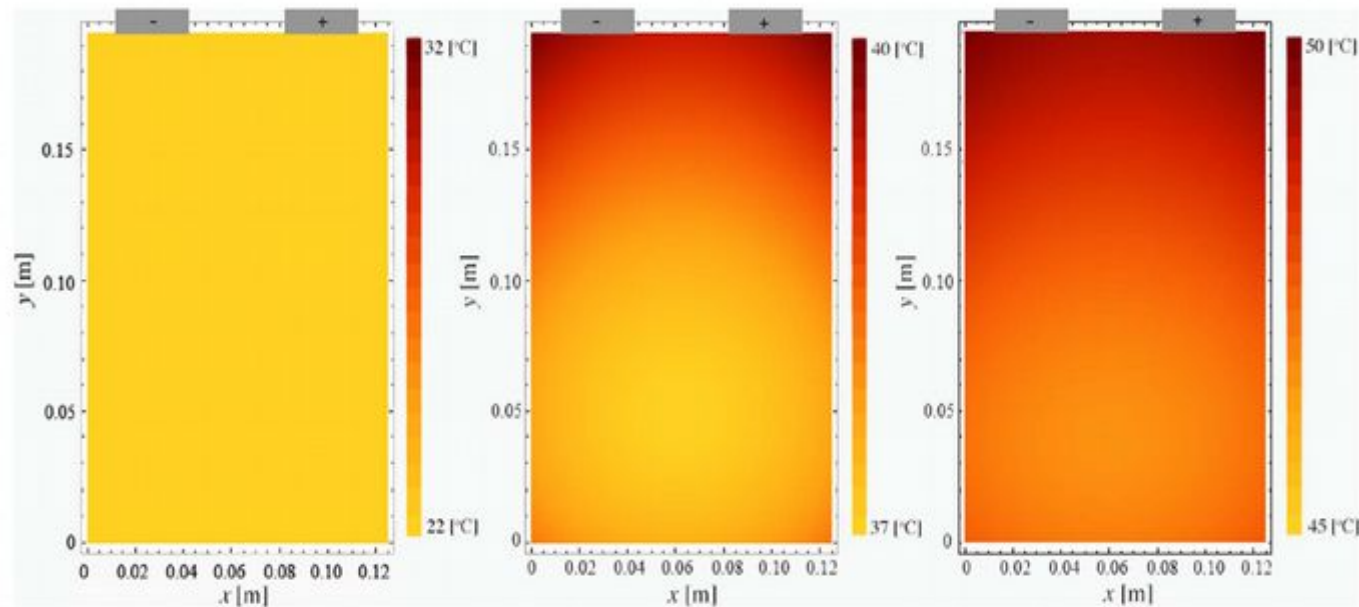
Electrochemical processes during normal charge / discharge cycle

For high-current applications, the irreversible heat generation dominates particularly during discharge

For a long cycle life, it is important to operate the cell in its optimum working range. Too low SOC. causes damage, as well as too high.

Heat Generation

Irreversible heat generation in the cell at high current discharge is not equally distributed, but localized near the terminals.



0% discharge

50% discharge

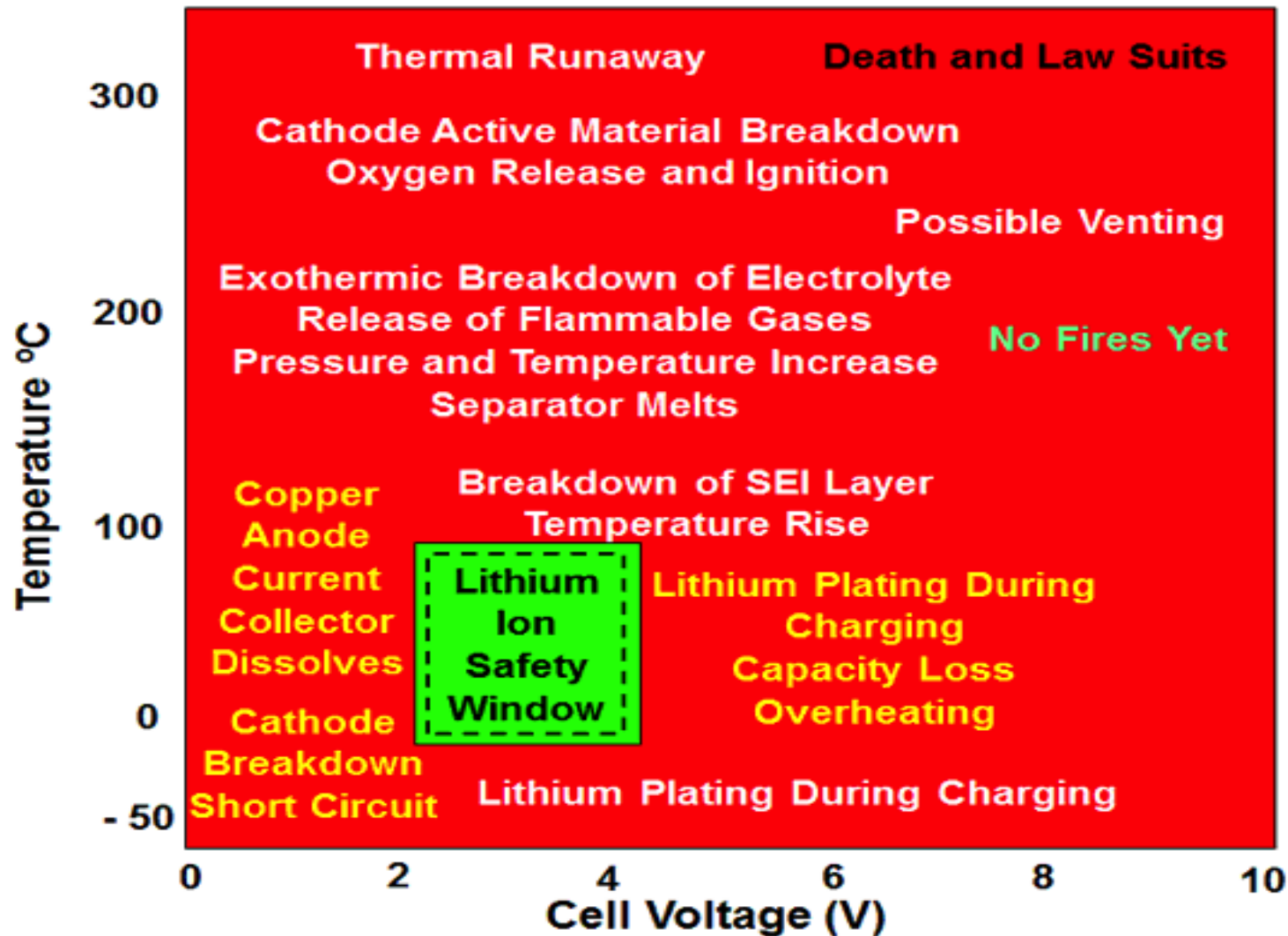
90% discharge

- cells do not age uniformly, there is a higher load area
- cell packs do not age uniformly as internal cells cannot dissipate their heat as well

Quelle: A Distributed Analytical Electro-Thermal Model for Pouch-Type Lithium-Ion Batteries, Maryam Yazdanpour,^a Peyman Taheri,^a Abraham Mansouri,^b and Majid Bahramia *Laboratory for Alternative Energy Conversion (LAEC), Journal of The Electrochemical Society*, 161 (14) A1953-A1963 (2014), pg 1962
(Thermal measurements of 60A Discharge for a 20Ah Lithium polymer battery)

Application window

Lithium Ion Cell Operating Window



Cell Chemistry

Positive electrode (cathode):

Lithiated form of a transition metal:

(eg, Lithium Cobalt (III) oxide LiCoO_2 , Lithium Nickel Oxide LiNiO_2 or Lithium Manganese oxide LiMn_2O_4) and mixed forms.

Negative electrode (anode): Carbon (C), usually graphite (C_6)

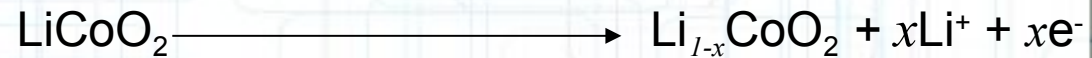
Electrolyte: Lithium salts (LiPF_6 , LiBF_4 , LiClO_4)

and organic solvents (Ethylene Carbonate, Dimethyl Carbonate, etc.)

Cell Chemistry

- Chemical reaction (charging)

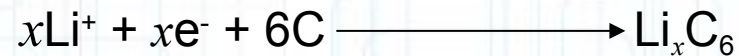
- Cathode



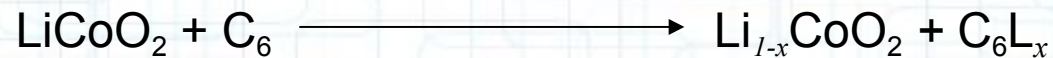
Inner Ion Migration

Exterior Electrical Current

- Anode



- General



Cathode loses lithium during charging, the anode is filled with lithium.

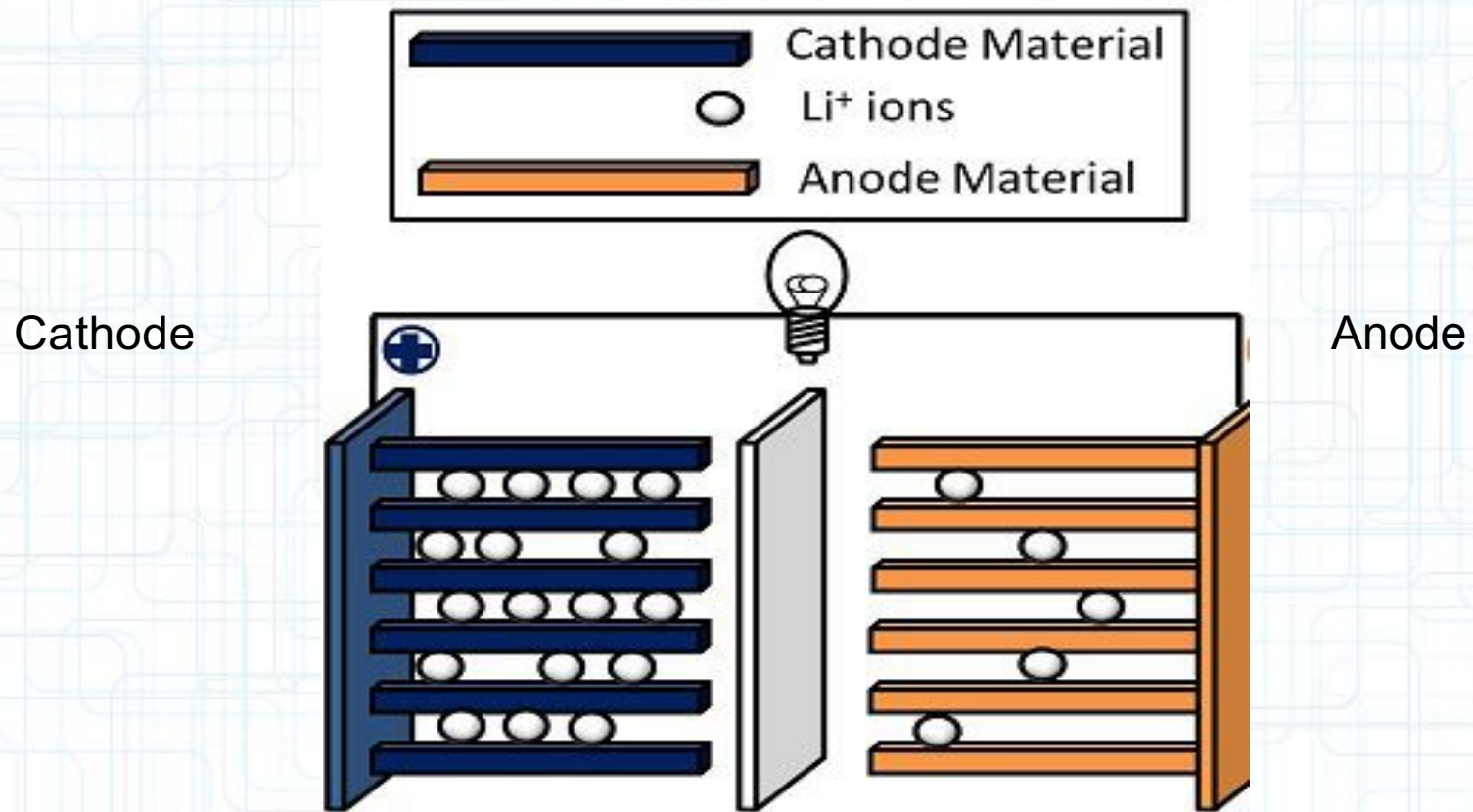
Cell Chemistry

Intercalation

Intercalation (from the Latin intercalare = insert)

In the chemical sense, is defined as the incorporation of molecules, ions (rarely atoms) in chemical compounds, which do not substantially alter their structure during the storage process.

Cell Chemistry



Quelle: Wikipedia

Cell Chemistry

The cell performance depends on: Current: Ion transport capacity

"How many ions can flow per unit of time?" → High Current Cell Limited

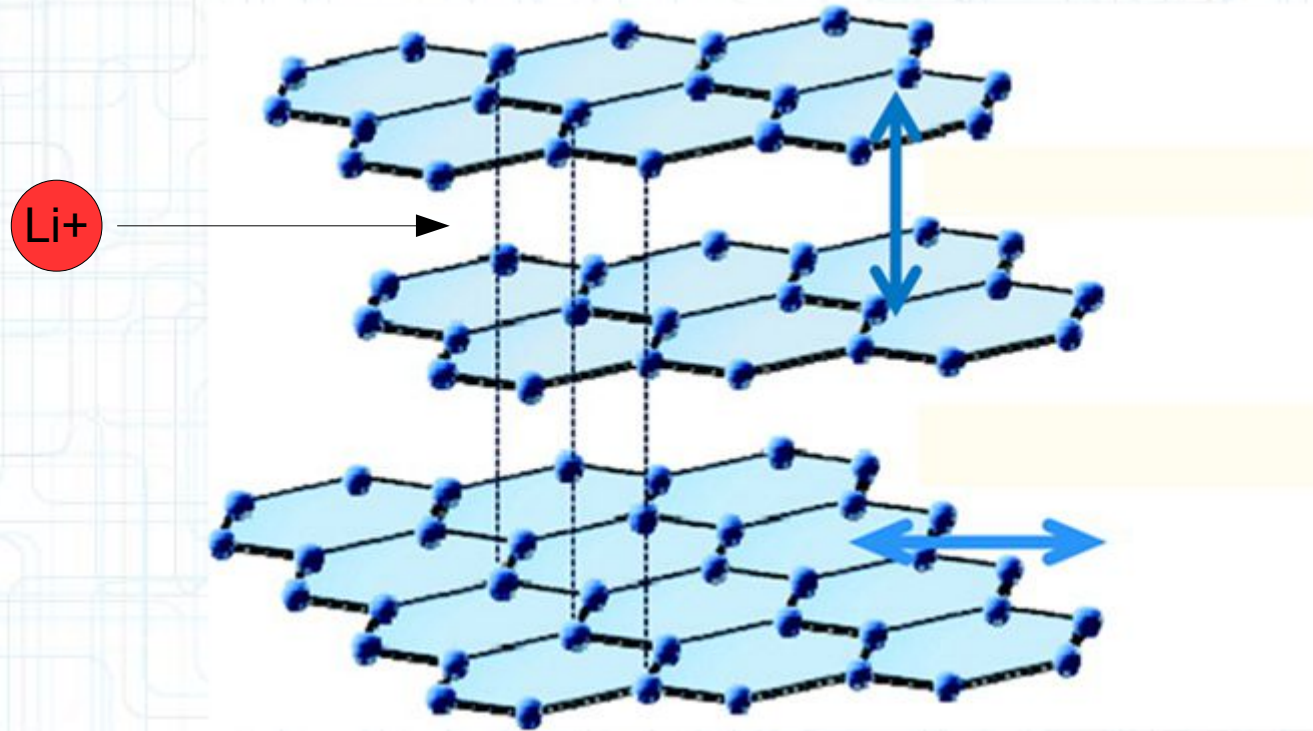
by: → Surface → Interfaces (Transitions / Layers) → Terminals →

Temperature Capacity: Ion release and ion absorption capacity

"How many ions, that the cathode emits during charging, can fit in the anode?" → High capacity cell

Cell Chemistry-Anode

The graphite anode consists of layers



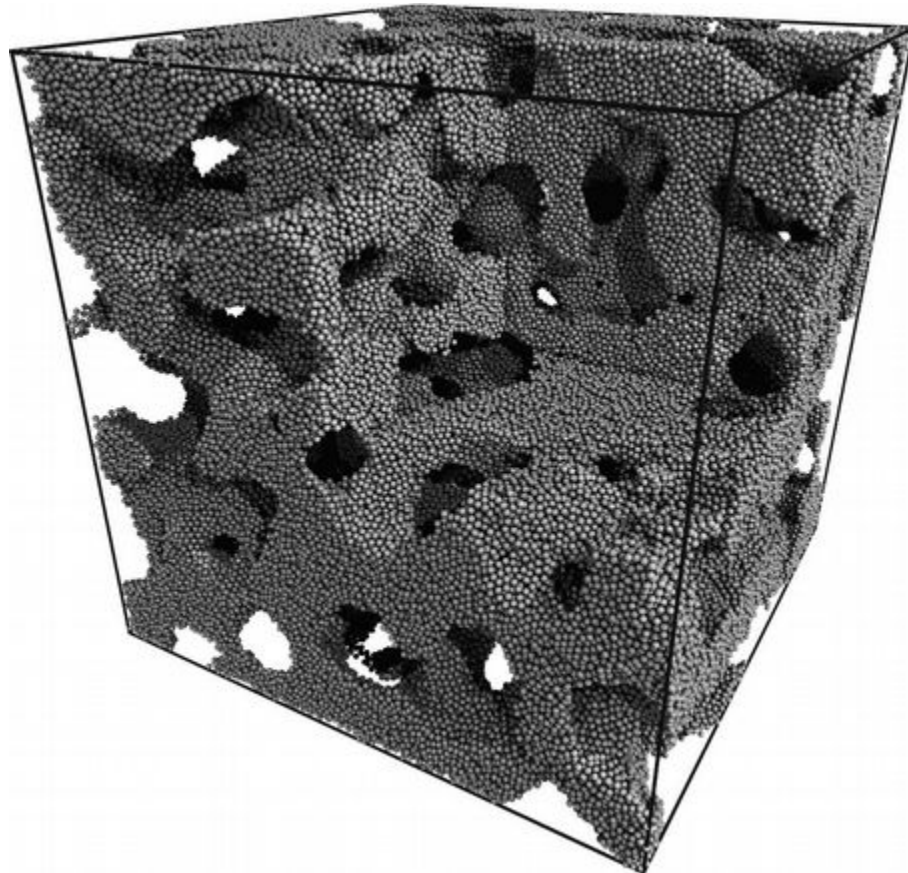
Ions can only penetrate from the side

→ Charging rate generally lower than discharge
Inner layers are more deprived of ions than the outer layers

Cell Chemistry-Anode

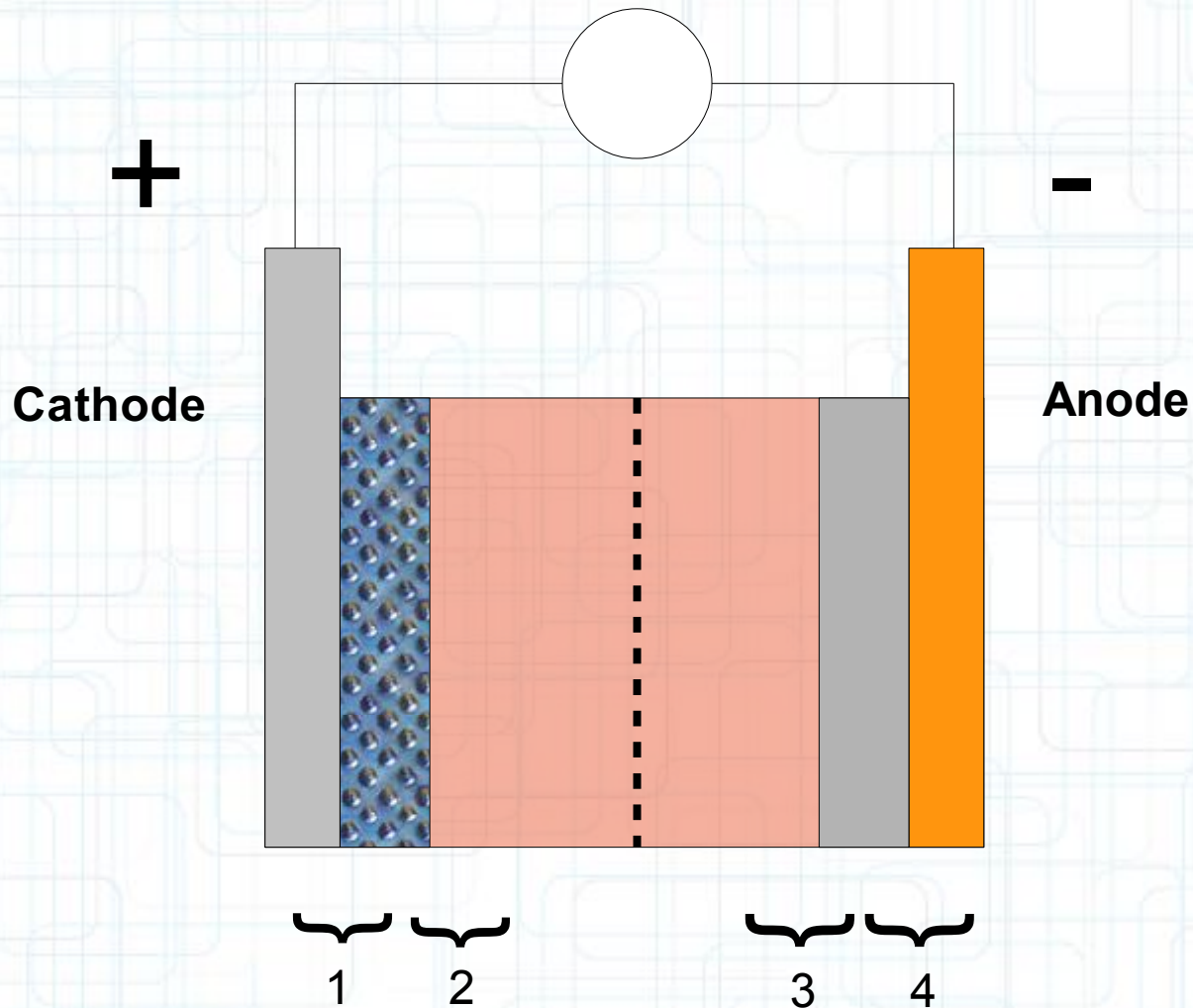
Nanotechnology:

Graphite layers consists of a finer "nanoporous structure"
The nanoporous graphite structure collectively has a larger surface area



Nanoporous carbon: source ncsu.edu

Transition Zones (Interfaces)

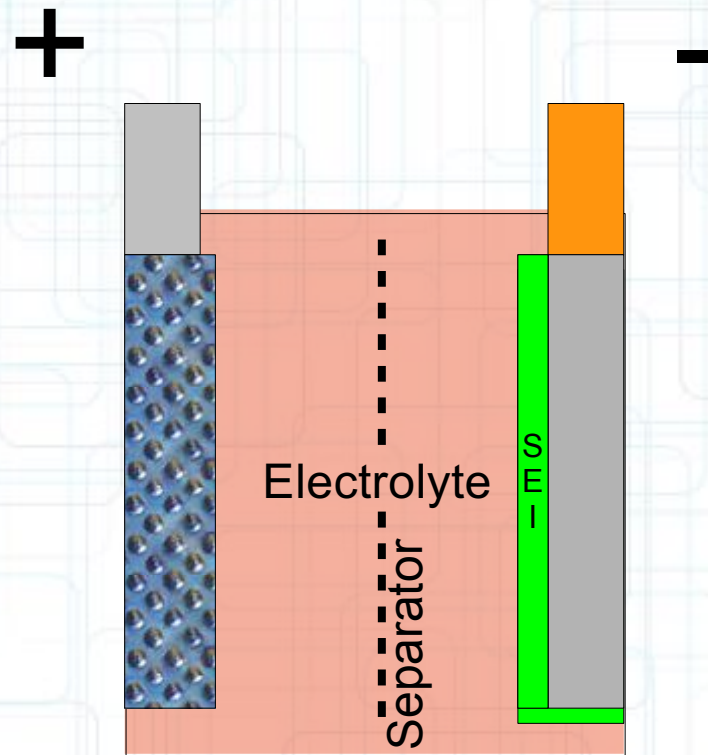


Most relevant are: Zone 2: "cathode - electrolyte" and 3 "electrolyte - anode",
The latter particularly is extremely important for the function of the cell.

Cell Chemistry-Anode

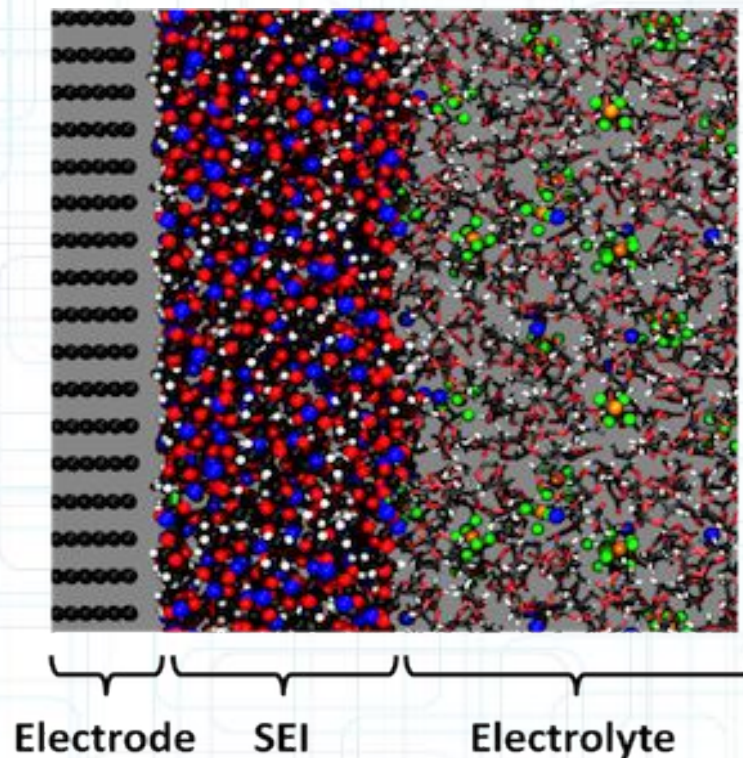
The graphite anode would be destroyed by reactions with the electrolyte.
To stop this process a protective coating has to be applied on the carbon.
This layer is called the:

Solid Electrolyte Interphase (SEI)



Cell Chemistry -Anode

The SEI layer is composed of the electrolyte and the anode in the initial formation (first charge during the manufacturing process). Lithium ions must penetrate it during the charging and discharging.



Quelle: 'A snapshot of the interfaces between the graphitic anode, a representative SEI, and the electrolyte.' The University of Chicago, Searle Chemistry Laboratory, 2012, Martin McCullagh, Ruibin Liang, <https://vothgroup.uchicago.edu/research/renewable-energy-materials>

Cell Chemistry - Anode

SEI-Layer:

Protection and stabilization of the anode

Without an SEI there is no cycle stability

in addition to this phenomenon

Increase in internal resistance

The thicker the SEI layer the higher the resistance

Subsequent loss of free lithium

The thicker the layer the less the remaining capacity

Decreased lithium migration

The thicker SEI layer the more sensitive to plating deposit

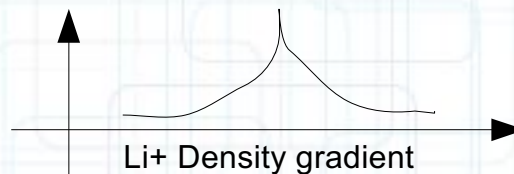
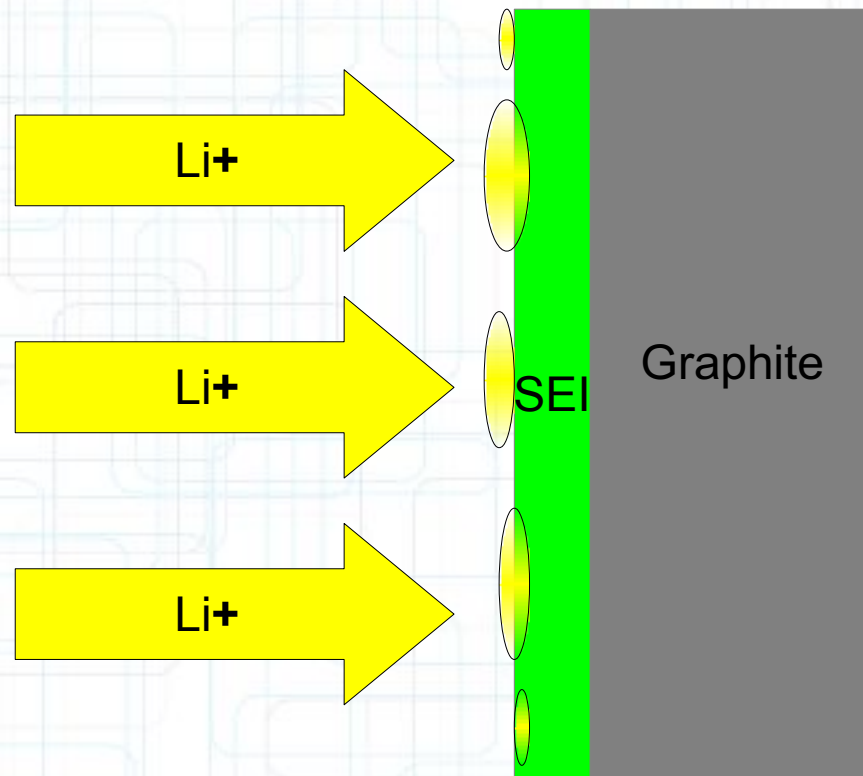
Cell Chemistry- Anode

The SEI layer is basically constructed in the preparation of the cell. Any cell which is 'new' has already experienced at least one preparation cycle.

The SEI layer grows during the life of the cell, the more degradation products caused in the cell, the thicker it is. High pressure and temperature accelerate this.

Cell Chemistry- Anode

Plating: Deposition of metallic lithium at the anode when charging
 Li^+ Density gradient

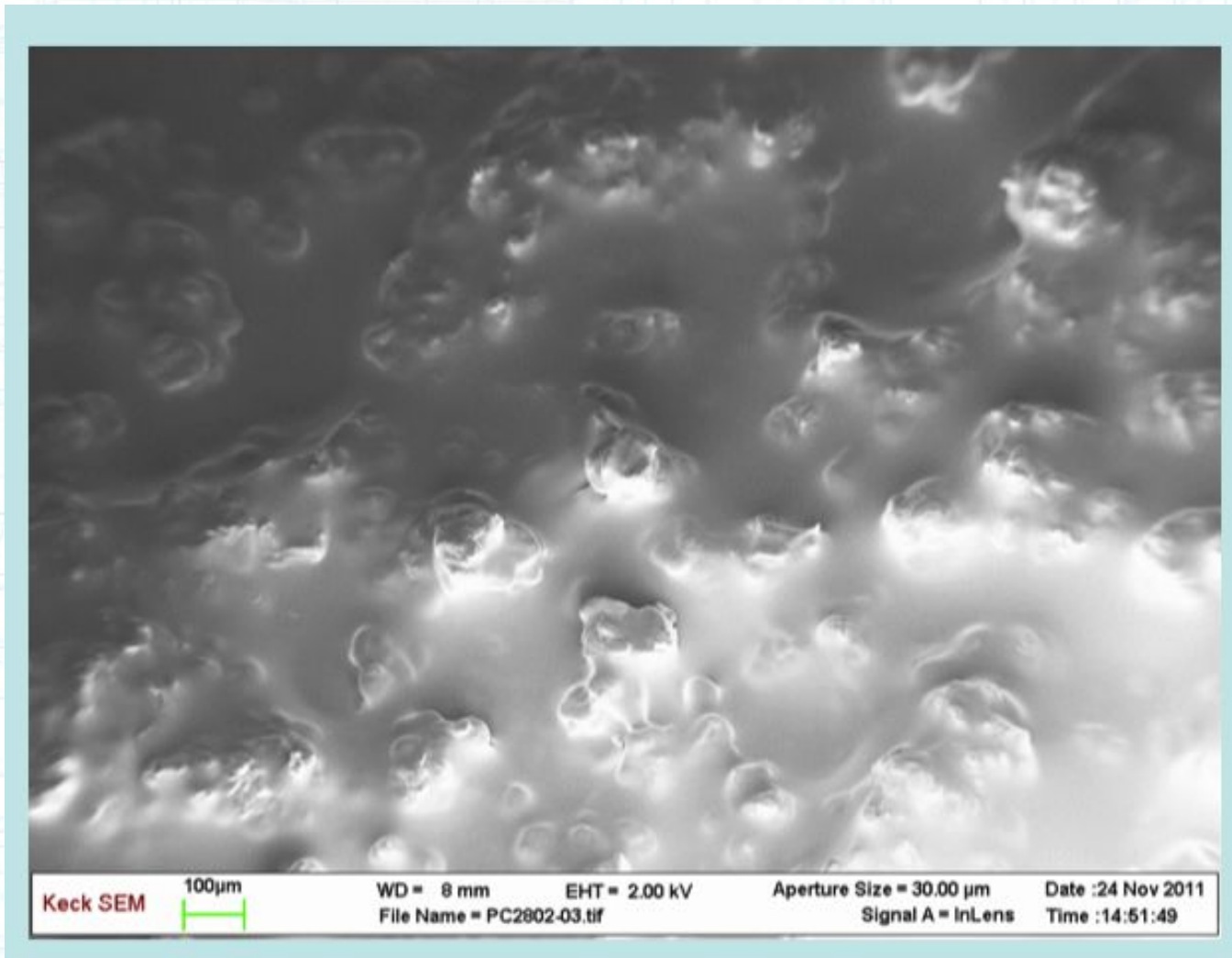


Cell Chemistry - Anode

Plating occurs when 'the anode wants more Li + ions in' than can be accommodated - and transportation capacity is available.→When charging, not during discharge
Reaches maximum capacity: Overload, already fully replenished anode(> 4.2V in normal cells)
Reaches transport capacity:- too high charging current
- depending on temperature (the lower the temperature, the worse it is)- depending on cell age (deposits oxidation products of the electrolyte) and SEI Layer thickness

Cell Chemistry - Anode

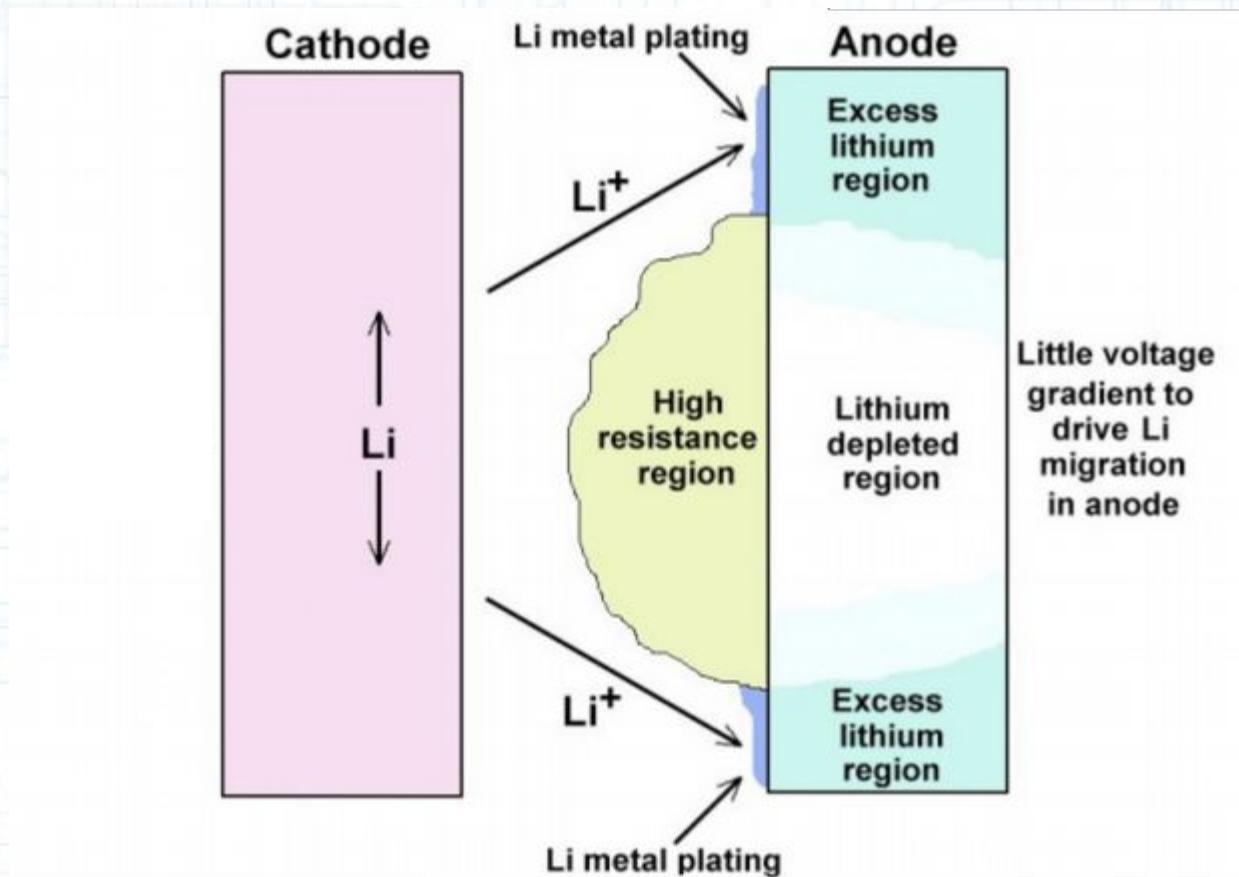
Li plating forms not in clean 'layers' but in "towers" (dendrites)



Quelle: Archer Group - Cornell University, 2011

Cell Chemistry - Anode

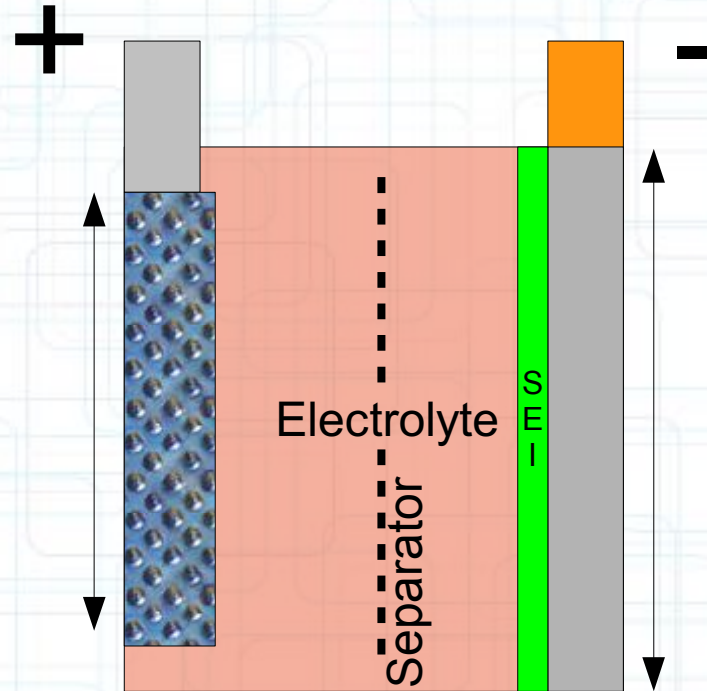
Plating is also caused by "flaws"



Quelle: Lithium Plating in Lithium-Ion Cells, Albert H. Zimmerman and Michael V. Quinzio
The Aerospace Corporation, Presented at the NASA Battery Workshop 16-18 November 2010, pg. 9

Cell Chemistry - Anode

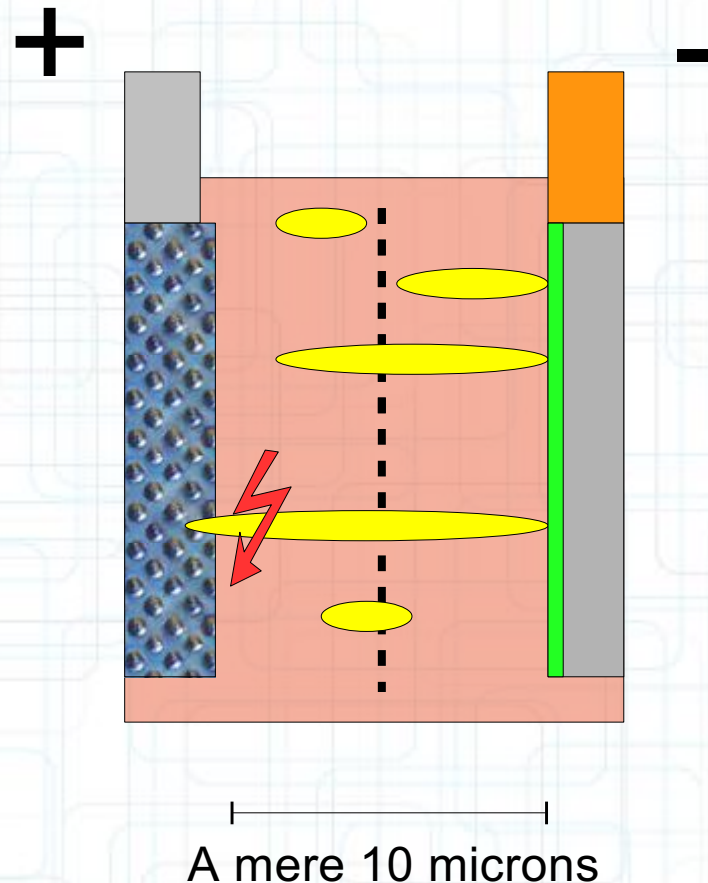
Also, in production, the anode is designed always somewhat larger than the cathode otherwise at the edge zones plating would occur.



This excess is referred to as "Overhang"

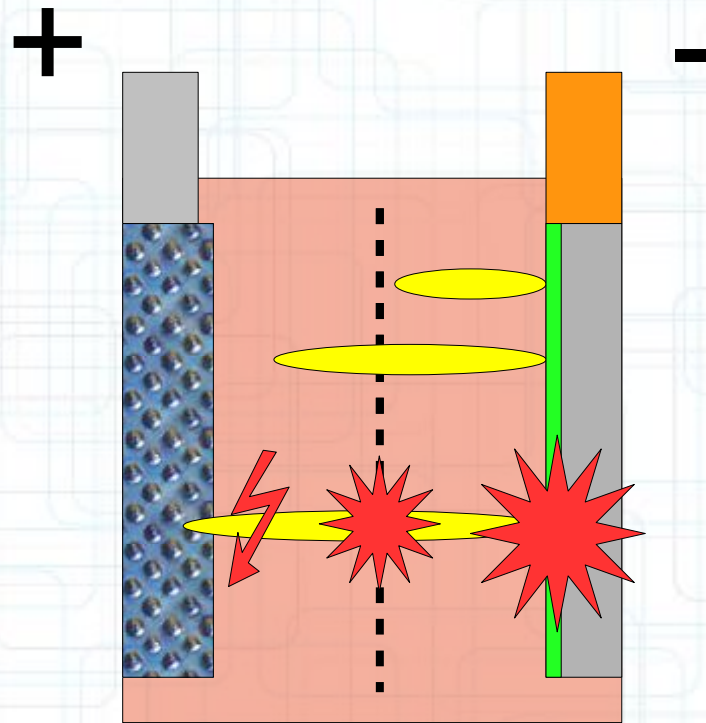
Cell Chemistry - Anode

Plating causes: Lithium loss / Li dendrite
If separator is bridged: "A Micro Short senario"



Cell Chemistry - Anode

Micro Shorts" can be "Macro Shorts":



Extreme local temperature increase- Separator is damaged

- Local collapse of the SEI layer: Exothermic!
- This resulting defect leads to further plating

Cell Chemistry - Runaway

Self-heating rate $> 10^{\circ}\text{C} / \text{minute}$

From about $150^{\circ}\text{C} \rightarrow$ Thermal Runaway

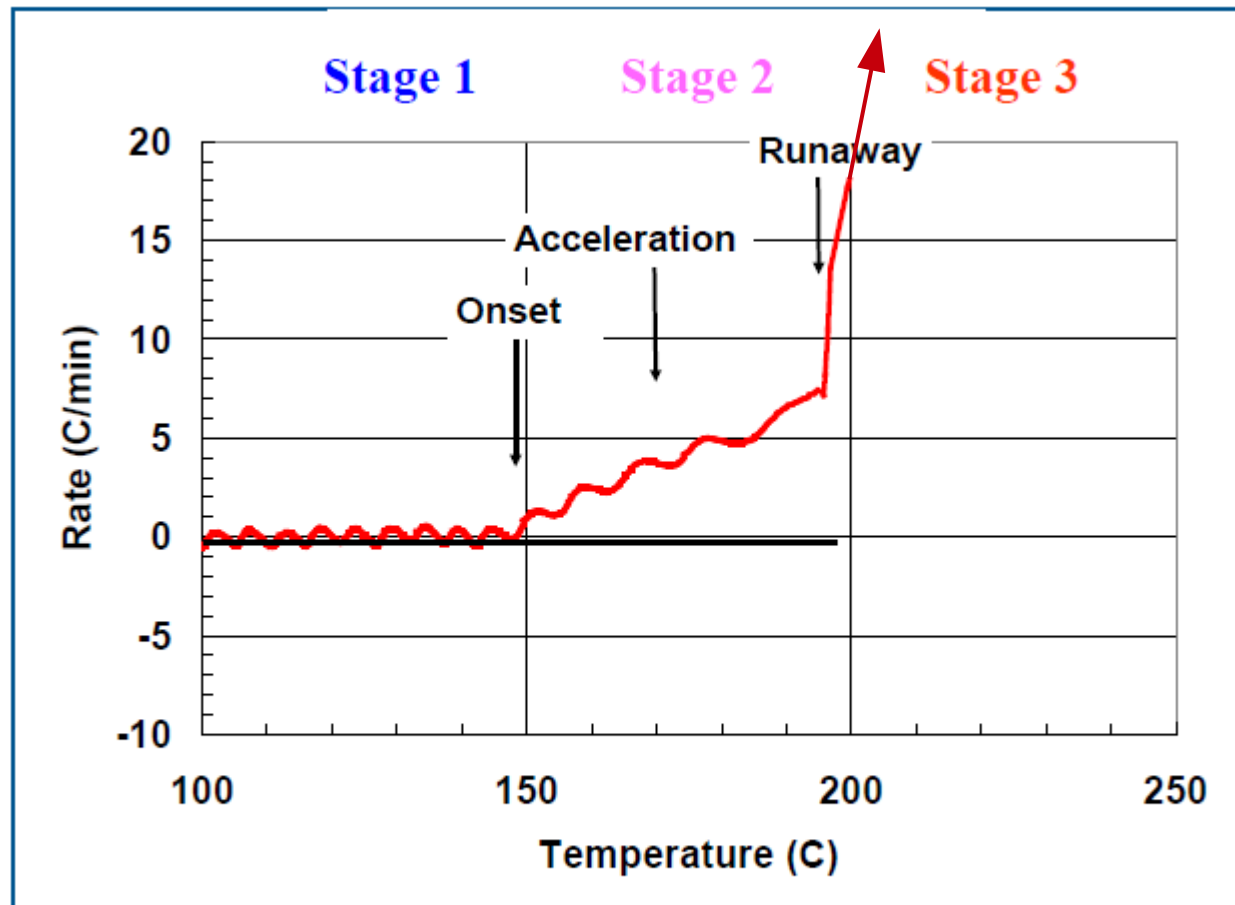
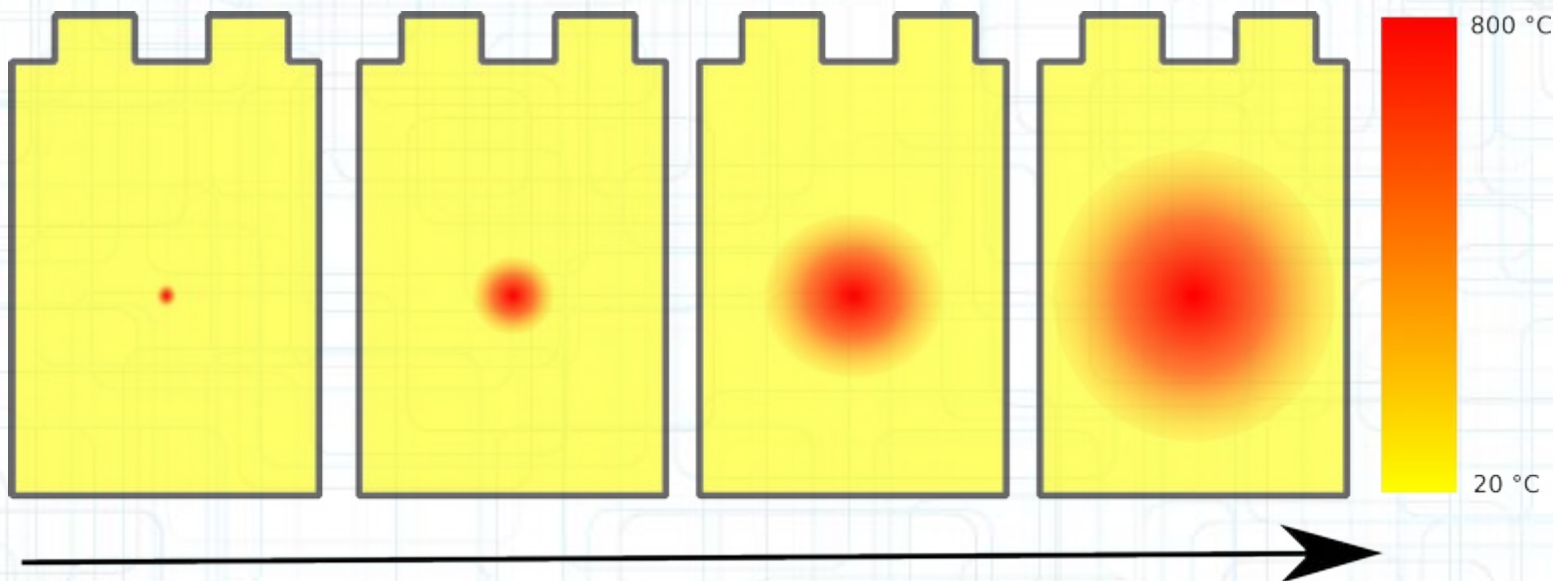


FIG. 3. Cell self-heating rate during forced thermal ramp test of Li-ion Gen 2 chemistry: anode = MCMB | electrolyte = 1.2 M LiPF_6 in EC:PC:DMC | cathode = $\text{LiNi}_{0.8}\text{Co}_{0.05}\text{Al}_{0.05}\text{O}_2$ | separator = Celgard 2325 trilayer. (From Ref. 10)

Quelle: A general discussion of Lilon Battery safety, Dan Doughty, E. Peter Roth, The electrochemical society Interface 2012. pg 39 (Roter Pfeil eingefügt)

Cell Chemistry - Runaway

Runaway starts around 150 ° C. The heat build-up to the 'Onset' is primarily based on resistive heating by the "Short", to about 250° C then it becomes a significant exothermic decomposition processes. If the runaway temperature is reached locally and the affected area is sufficiently large it spreads very quickly in the entire cell.

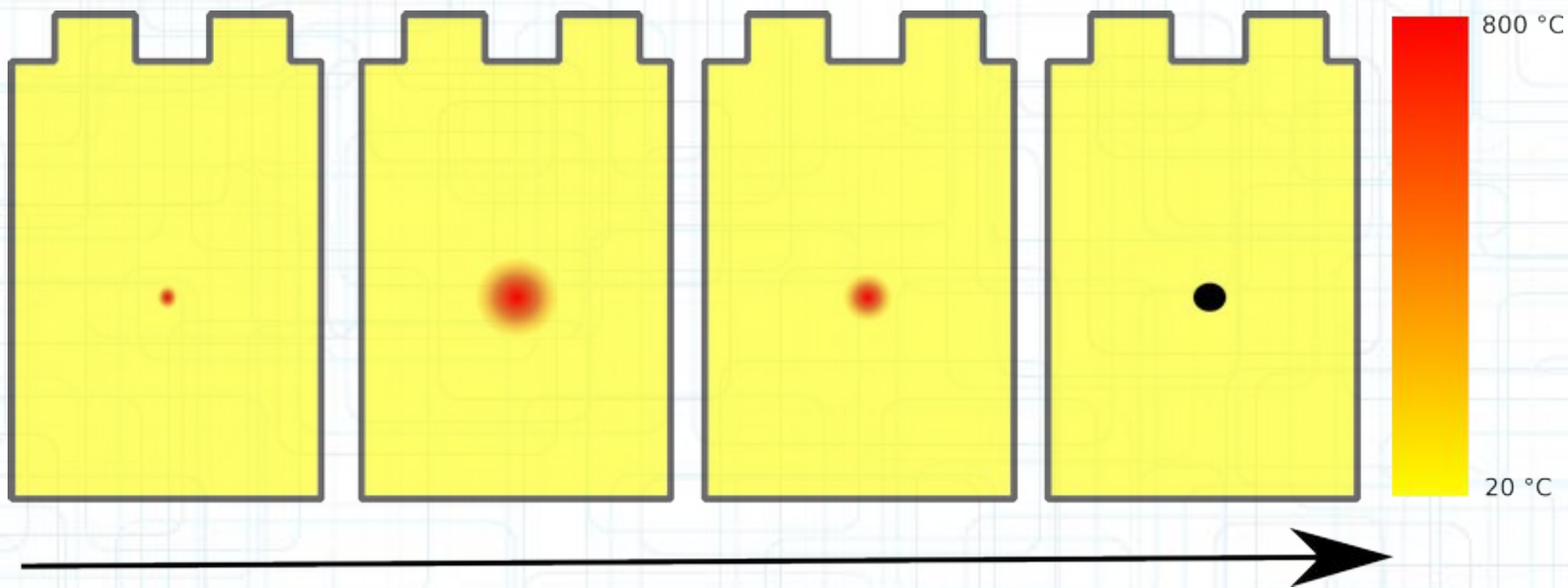


Cell Chemistry - Runaway

If there is not sufficient heat generation, then the result is only a void.

The self-limitation occurs in part because the separator expands when heated and thereby locally lowers its ion conductivity.

As long as the separator does not melt, the short circuit will be limited. However, this faulty point is then possibly again a trigger for further plating.



Cell Chemistry - Runaway

The temperature rise to 'Onset' can also occur slowly (minutes to hours)

Once this is achieved, however, the heating occurs faster locally.

The warming 'Onset' gets its energy from the charging of the cell.

In a cool storage environment, a higher heat generation is necessary.

This also assumes the necessary size of the triggering 'Shorts'.

Bigger 'Shorts' are rare and reach a certain size only by mechanical damage.

From outside these processes in the cell are not easy to detect.

For a small cell capacity this damage can be detected from a slightly elevated self-discharge. For larger capacities, however, this indication is weaker.

In such cells ($<3.3\text{V}$) problematic cells can only be detected in test runs by repeated slow charge / discharge in the low cell voltage range.

On the basis of small, impulsive changes the charging current can be detected.

→ Micro-shorts cannot easily be ruled out, so you have to minimize the likelihood they trigger Runaways.

Cell Chemistry - Runaway

- Cell must have sufficient energy stored, the higher the SOC the more likely it is
- High ambient temperature favours Runaway
 - **Cool (<20 ° C) and store "no charge"**

Cell Chemistry - Anode

Factors for the formation of plating:-

High charging current - Low temperatures
(while discharging)- Old /overloaded cells
(thick SEI layer)

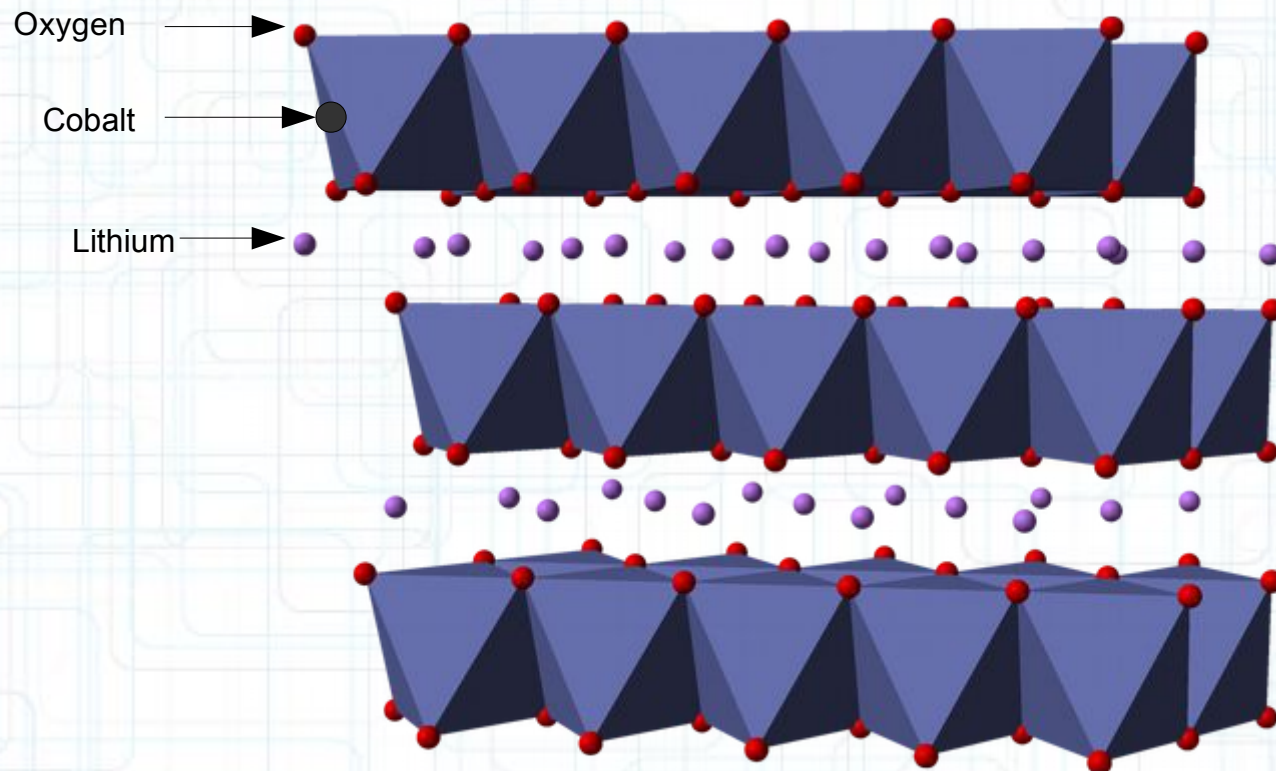
- Flaws (manufacturing defects / damage)
- Too high discharge current

See explanation of the Cathode.

→ Never cold discharge cells at a high C-rate load.

Cell Chemistry - Cathode

Cathode consists of Lithium Metal Oxides, such as LiCoO_2



Quelle: Wikimedia.org

Cell Chemistry - Cathode

- When charging Li^+ ions move from the cathode
- Upon discharge the cathode accepts Li^+ ions
- As with the anode, there is a transition zone ("interface") to the electrolyte
- In contrast to the anode there is no "SEI layer" but instead reaction products diffuse into the electrolyte

Cell Chemistry - Cathode

At high discharge rates, the cathode is under mechanical stress.

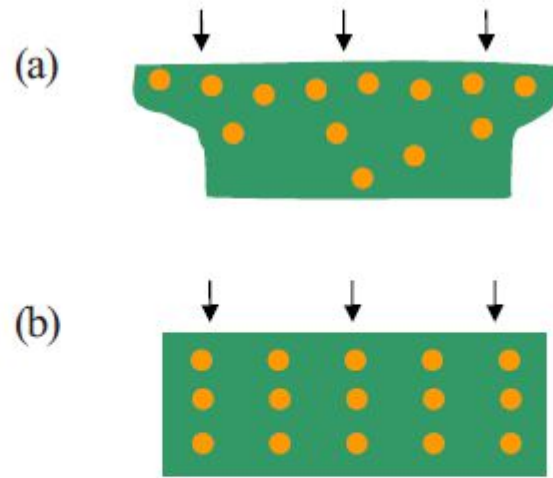
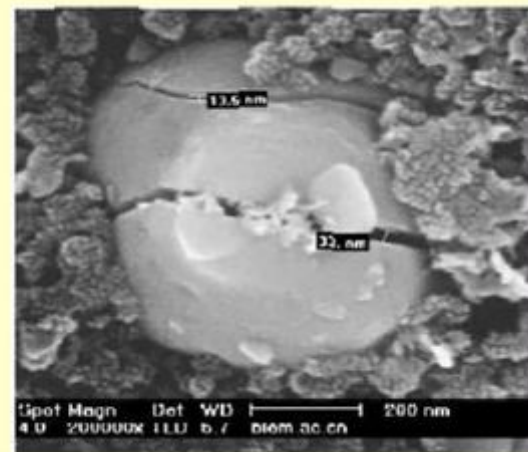
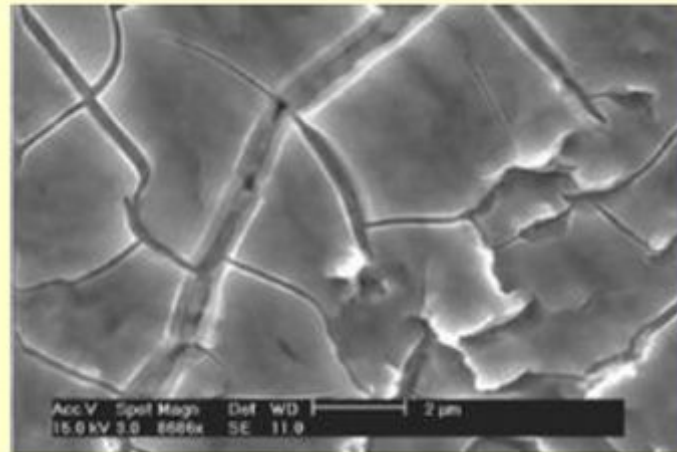


FIG. 2. (Color online) (a) When the rate of discharging is high, the distribution of lithium in the active particle is inhomogeneous, which causes a field of stress in the particle. (b) When the rate of discharging is low, the distribution of lithium in the particle is negligible. The arrows indicate the direction of lithium insertion.

Cell Chemistry - Cathode

High discharge rates, especially at low temperatures lead to cracks and fractures in the Cathode

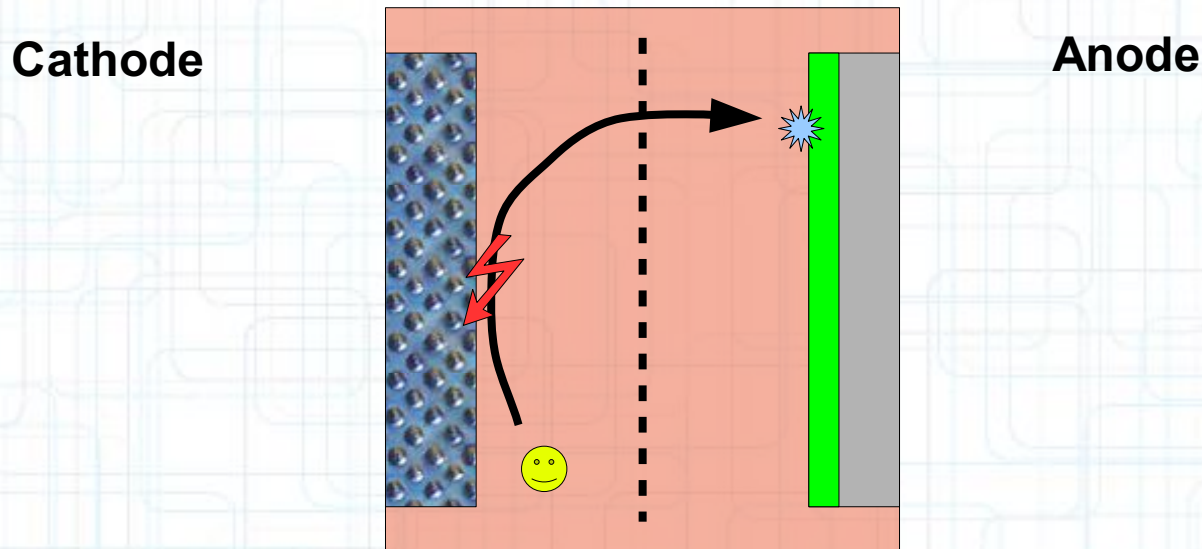


Quelle: Stress and Deformation Caused by Insertion in Li-ion Batteries,
Department of Physics, Harvard University, Cambridge, MA 02138, USA

→ Never overload cold cells!

Cell Chemistry - Cathode

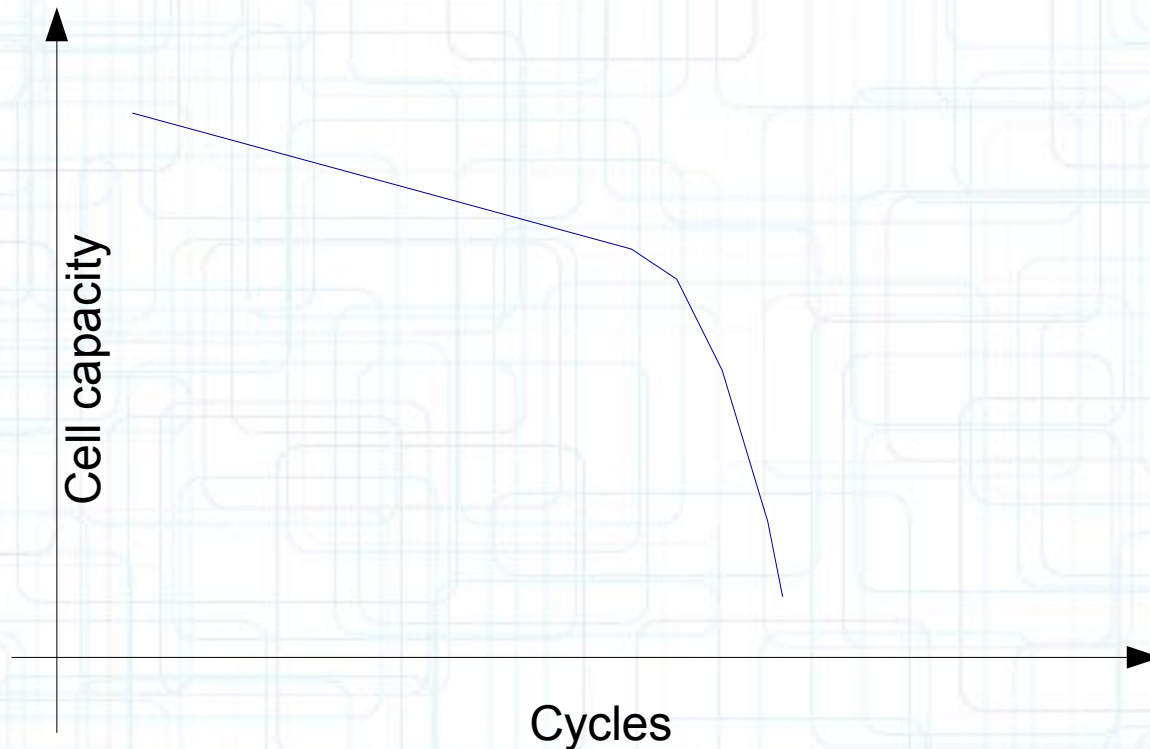
Too high temperatures - even locally - and/or cell voltage ($> 4.1\text{V}$) cause reaction processes (oxidation) of the electrolyte at the cathode layer.



The reaction products then are deposited primarily on the SEI at the anode → increasing internal resistance + loss of capacity

These reactions also produce gases (mostly CO_2), the main reason for irreversible "puffing" after overload or storage at fully charged state (esp. at high temperatures).

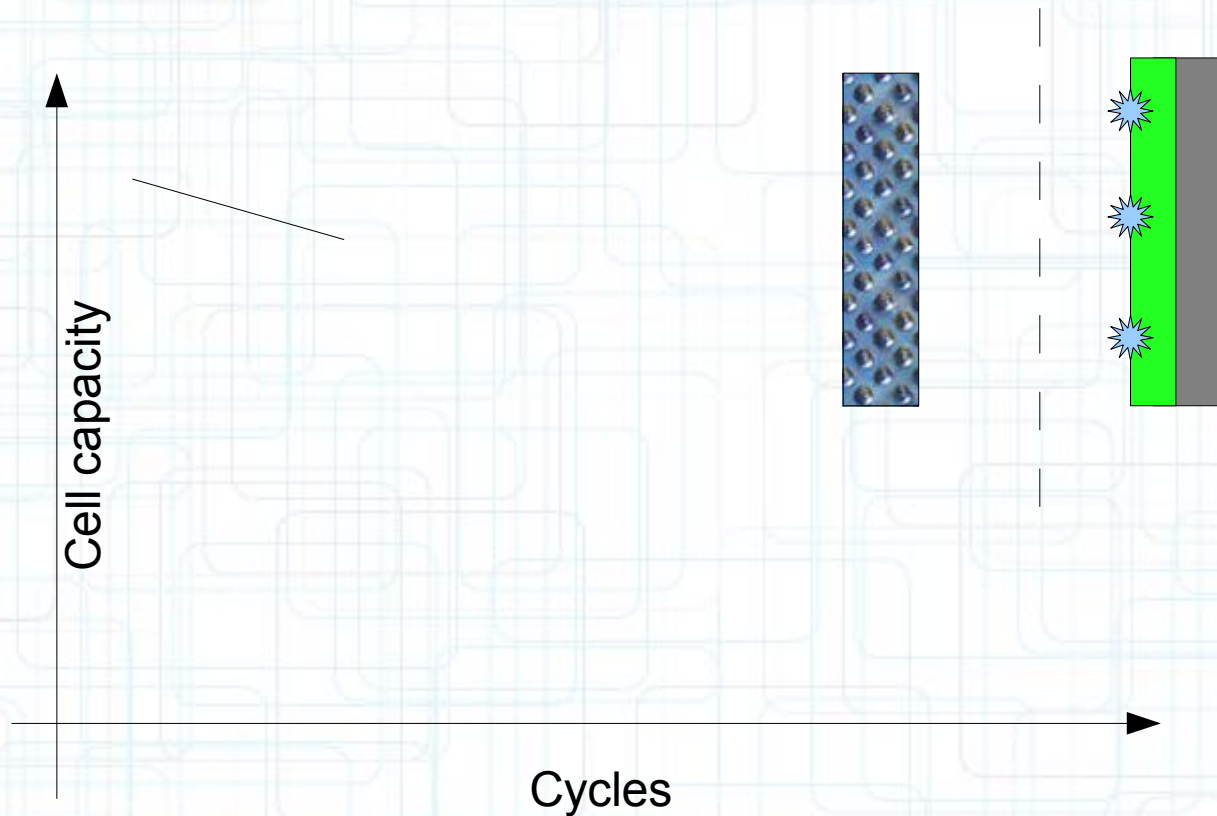
Cell Chemistry - Cathode



Accumulation of oxidation products at SEI Layer is the main reason for 'kink' in the loss of capacity curve

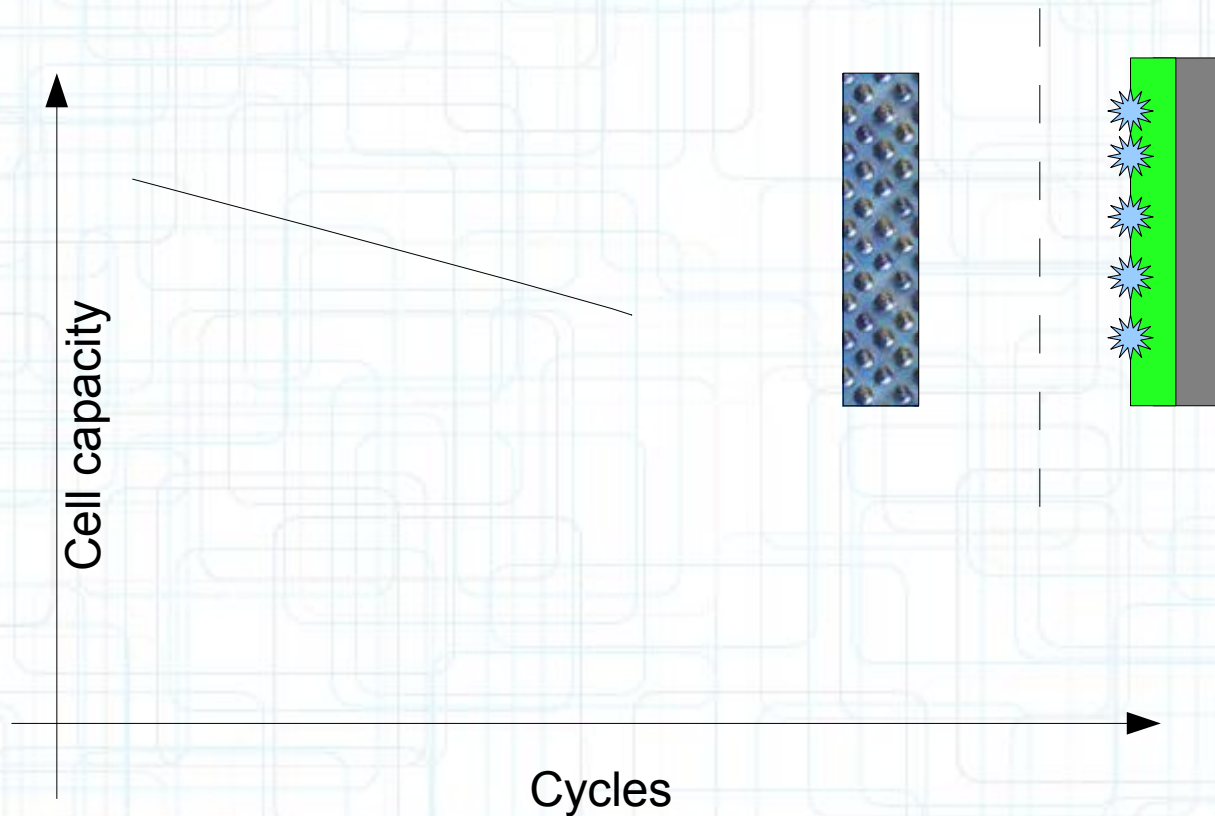
Goes hand in hand with an increase in the associated internal resistance)

Cell Chemistry - Cathode



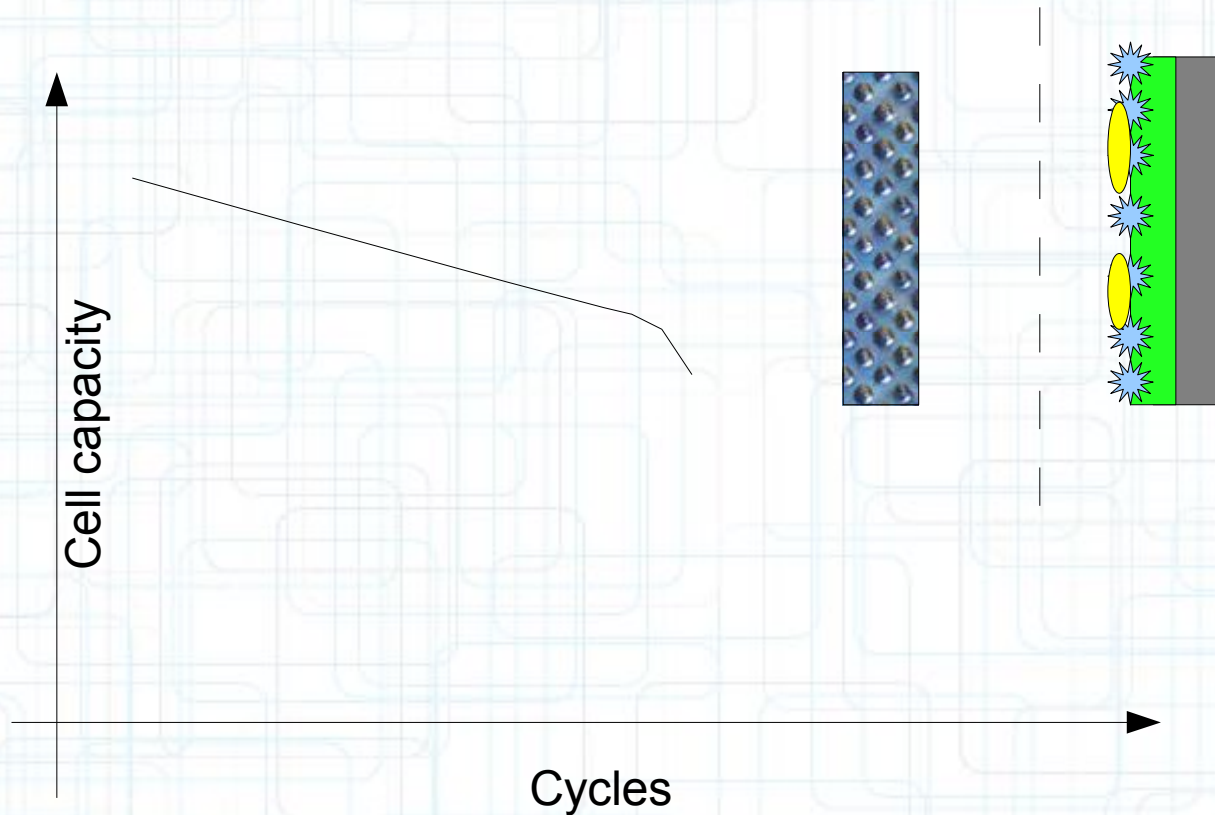
Initially there is minimal effect, the SEI surface is sufficient

Cell Chemistry - Cathode



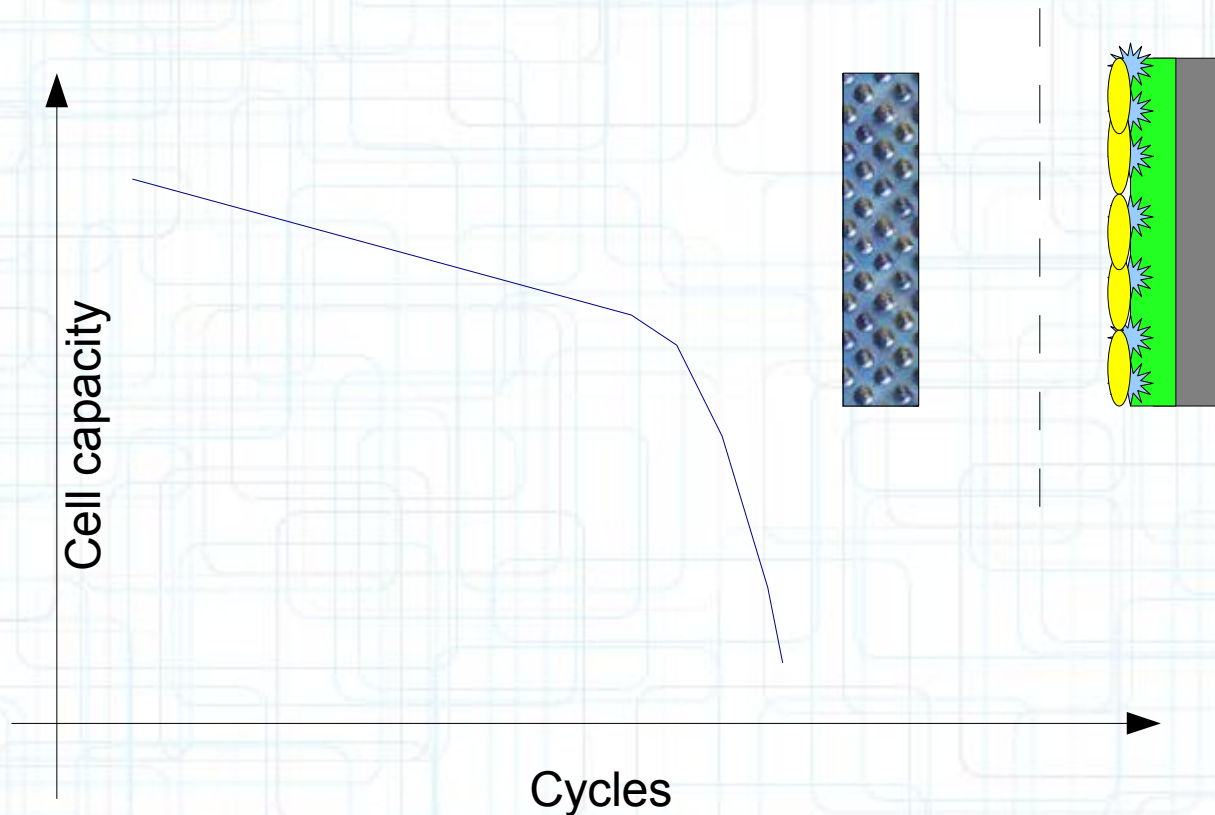
Increasing effect, SEI surface still just enough

Cell Chemistry - Cathode



Start of accelerated aging, internal resistance increased significantly, plating sets in, lithium loss is relevant.

Cell Chemistry - Cathode



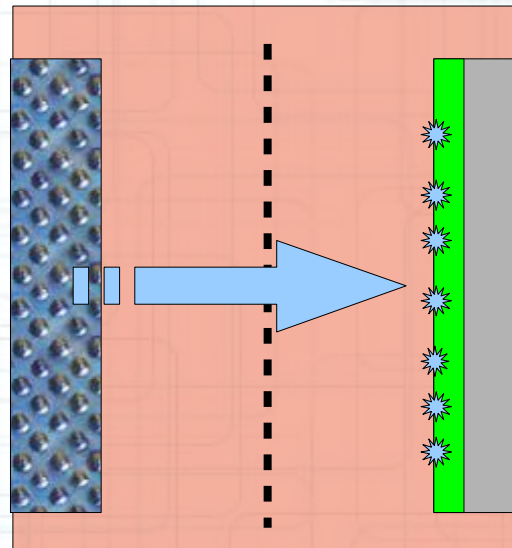
Plating is dominant, Lithium loss high. Cell clearly loses capacity.
Strongly increasing internal resistance. Therefore:-

→ **Do not overload/overburden the cells**

Cell Chemistry - Cathode

Excessive temperatures lead to the release of free "metals" at the cathode.

Cathode



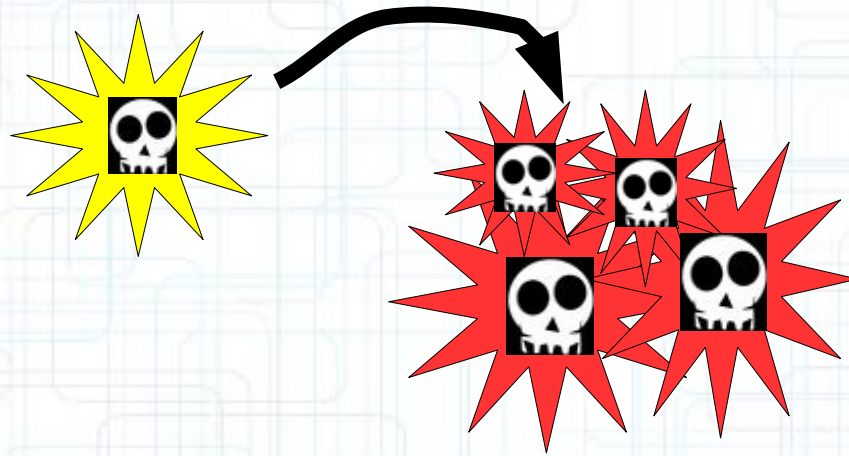
Anode

→ also causes "poisoning" of the SEI layer

This is a problem mainly in LiFePO_4 cells → triggering iron deposition at temperatures $> 45^\circ \text{C}$

→ **Do not store cells in a warm environment!**

" Memory Damage "



Cell damage accumulates over the period of use.
A cell does not forget mistreatment!

Cell Chemistry - Electrolyte

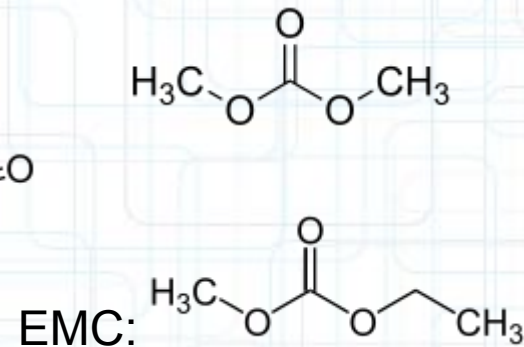
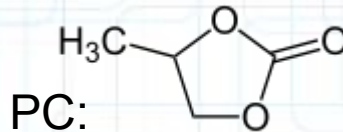
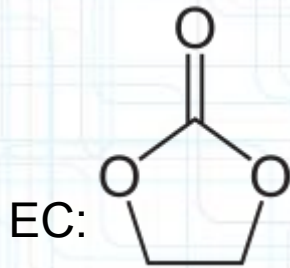
Liquid or gel

Three basic components:

Electrolyte salt" (LiPo): LiPF_6



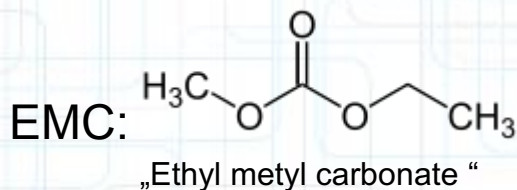
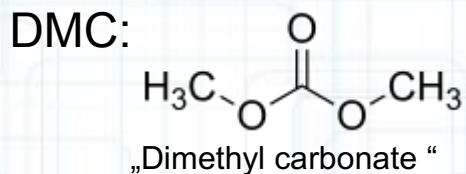
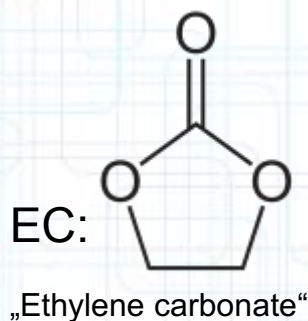
- Organic solvents, including EC: PC: DMC: EMC:



- Additive

Cell Chemistry - Electrolyte

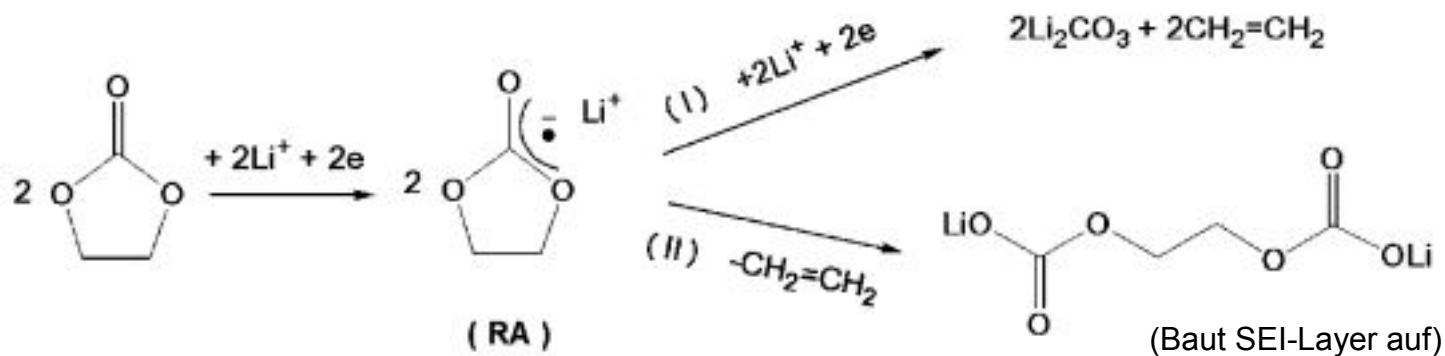
The mixing ratio of the organic solvent determines whether at room temperature it is more liquid or gel-like.



Simplified: The higher the EC share of the mixing ratio the more solid, and thermally stable at high temperatures the electrolyte is

Cell Chemistry - Electrolyte

Ethylene Carbonate (EC) is always included in the mixture as it is necessary for the formation of the SEI layer on the Anode.
(Builds the SEI layer on the Anode)



Quelle: A review on electrolyte additives for lithium-ion batteries, Journal of Power Sources 162 (2006) 1379–1394

EC $\xrightarrow{\text{Formed in the first cycle}}$ SEI-Layer

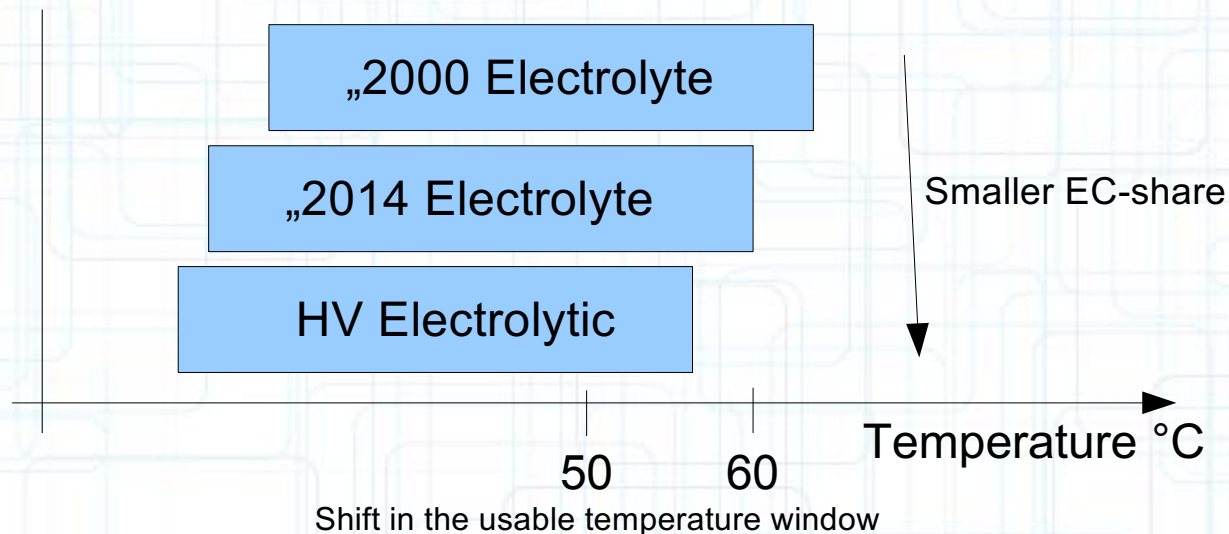
Cell Chemistry - Electrolyte

The mixing ratio determines the temperature and voltage capability.

EC / DMC 1: 1 electrolyte is unstable from about 4.25V

Compared: eg EC / DMC / diethyl (DEC) in a 3: 3: 4 ratio is stable to 4.5V.

The EC proportion has declined in recent years (From $\sim 50\% \rightarrow \sim 25\%$).
HV cells have smaller proportions of EC in the electrolyte and are therefore slightly less temperature stable



Cell Chemistry - Electrolyte

Depending on electrolyte composition the formation after the first charge is not yet fully completed.

A gentle treatment of the cell in the first few cycles may therefore be useful.

Electrolyte-Type	Rev.Cap mAh/g (1st Cycle)	Irr.Cap mAh/g (1st Cycle)	Rev.Cap mAh/g (5th Cycle)	Irr.Cap mAh/g (5th Cycle)
EC+DEC+DMC (1:1:1)	306.5	51.8	310.8	85.9
EC+DEC+DMC+MA (1:1:1:1)	201.5	36.9	236.5	56.9
EC+DEC+DMC+EA (1:1:1:1)	210.4	49.9	214.2	68.5
EC+DEC+DMC+EP (1:1:1:1)	233.4	49.06	340.75	88.30
EC+DEC+DMC+EB (1:1:1:1)	272.0	55.6	309.46	90.86

Reversible and irreversible capacities of graphite electrodes (Li-C cell) in contact with various electrolytes.

Quelle: The Role of Electrolyte Upon the SEI Formation Characteristics and Low Temperature Performance of Lithium-Ion Cells with Graphite Anodes, M. C. Smart, B. V. Ratnakumar, S. Greenbaum and S. Surampudi, Jet Propulsion Laboratory;
MA = methyl acetat, EA = ethyl acetat, EP = ethyl propionate, EB = ethyl butyrate, PC = propylen carbonate

Cell Chemistry - Additives

Improving the SEI formation- Protection of the cathode/electrolyte (Oxidation inhibition / Li Retention)

- LiPF₆ temperature stabilization
- Overload protection ("Redox Shuttle")
- Fire prevention ("Fire-Retardant")
- Wetting agent (Manufacturing, wetting of the electrode)
- Corrosion protection for the Aluminium

In 2014: About 3-5 additives in each cell



Failure

Voltage dependent:

Too high ($> 4.25\text{V}$):

Cathode:

Stress caused by
extreme delithiation

Anode:

Lithium-Plating
→ Li dendrite formation

Electrolyte:

Decay
(oxidation processes at
the cathode)

Too low ($< 2\text{V}$):

Cathode:

Stress due to high
delithiation,
Oxygen released

Anode:

Copper carrier dissolves in
released oxygen
→ Cu dendrite

Electrolyte:

Failure

Temperature dependent:

Too high ($> 60^{\circ}\text{C}$, $> 55^{\circ}\text{C}$ with newer cells):

<u>Cathode:</u>	<u>Anode:</u>	<u>Electrolyte:</u>
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Oxygen release

SEI breakdown.

Decay

Increased risk of thermal runaways!

Too low ($<15\text{-}20^{\circ}\text{C}$, high C-rate):

<u>Cathode:</u>	<u>Anode:</u>	<u>Electrolyte:</u>
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Stress from too
high Li⁺ flow,

Breakage / cracks
(during discharge)

Lithium-Plating
(during charge)

Failure

Load dependent (too high a current)

Too high (Discharge)

Cathode:

Anode:

Electrolyte:

Oxidation of electrolyte
(especially at high temp)

Accumulation of
degradation
products

Degradation

Stress by high Li⁺-Flow,
break / crack
(especially at low temp)

Too high (Charge, in particular at low temperature):

Cathode:

Anode:

Electrolyte

Plating

Failure

"Puffed" cells are divided into two different types:

"Reversible" puffing:

Occurs in high-current discharge, Reforms after cooling back down

- Basic: Evaporative components of the solvent in the electrolyte
- Signs of overload, but cell can still be used.

"Irreversible" puffing:

Does not reverse after cooling.

- Basic: Decay processes of the electrolyte form CO₂ and other gases.
- Indication of defects in the cell. Consequent loss of capacity and increased internal resistance can be expected.
- If a deep discharge occurred, immediately discard cell.

Probable damage to the cathode accompanied by oxygen release!

Failure

Damaged cells smell 'sweet' / 'fruity' if electrolyte leaks.
Such cells must be disposed of immediately.
Only hold with protective gloves,
as the conductive salt in the electrolyte is poisonous and corrosive.
Some additives are dangerous as well.

Conducting salts ("LiPo") LiPF₆

R34: Causes burns.

R24: Toxic in contact with skin.

R22: Harmful if swallowed.

Conducting salts ("LiFePo") LiBF₄

R20 / 21/22: Harmful by inhalation, and with skin contact.

R31: Contact with acids liberates toxic gas.

R34: Causes burns.

R36 / 37/38: Irritating to eyes, respiratory system and skin.

R23 / 24/25: Toxic by inhalation, in contact with skin.

R11: Highly flammable

Long Life

- Cool, dry and empty (3.7V / cell) storage ($<20^{\circ}\text{C}$)
- Bring up to operating temperature before charging and discharging.
- Never overcharge ($> 4.2\text{V}$ / cell), ($>4.25\text{-}4.35\text{V}$ / cell for HV)
- Never over-discharge ($<3.0\text{V}$ / cell) and if it occurs immediately recharge)
- Operating temperature $> 25^{\circ}\text{C}$, ideally $30 - 40^{\circ}\text{C}$
- Keep at working temperature only as long as absolutely necessary
- Do not operate overly warm ($<55\text{-}60^{\circ}\text{C}$)

Even longer life:

- Do not charge to 4.2V (cycle life is doubled if charged to just 4.1V *)
- Do not fully discharge, use shallow cycles ($<70\%$ DOD doubles lifespan *)
- Use low loads, do not load quickly (esp. at low temperatures)

* especially for low loads

Credits

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