# RELIB

## 30 Minute Review

6th November 2018

Paul Anderson, Andy Abbott, Bob Lee

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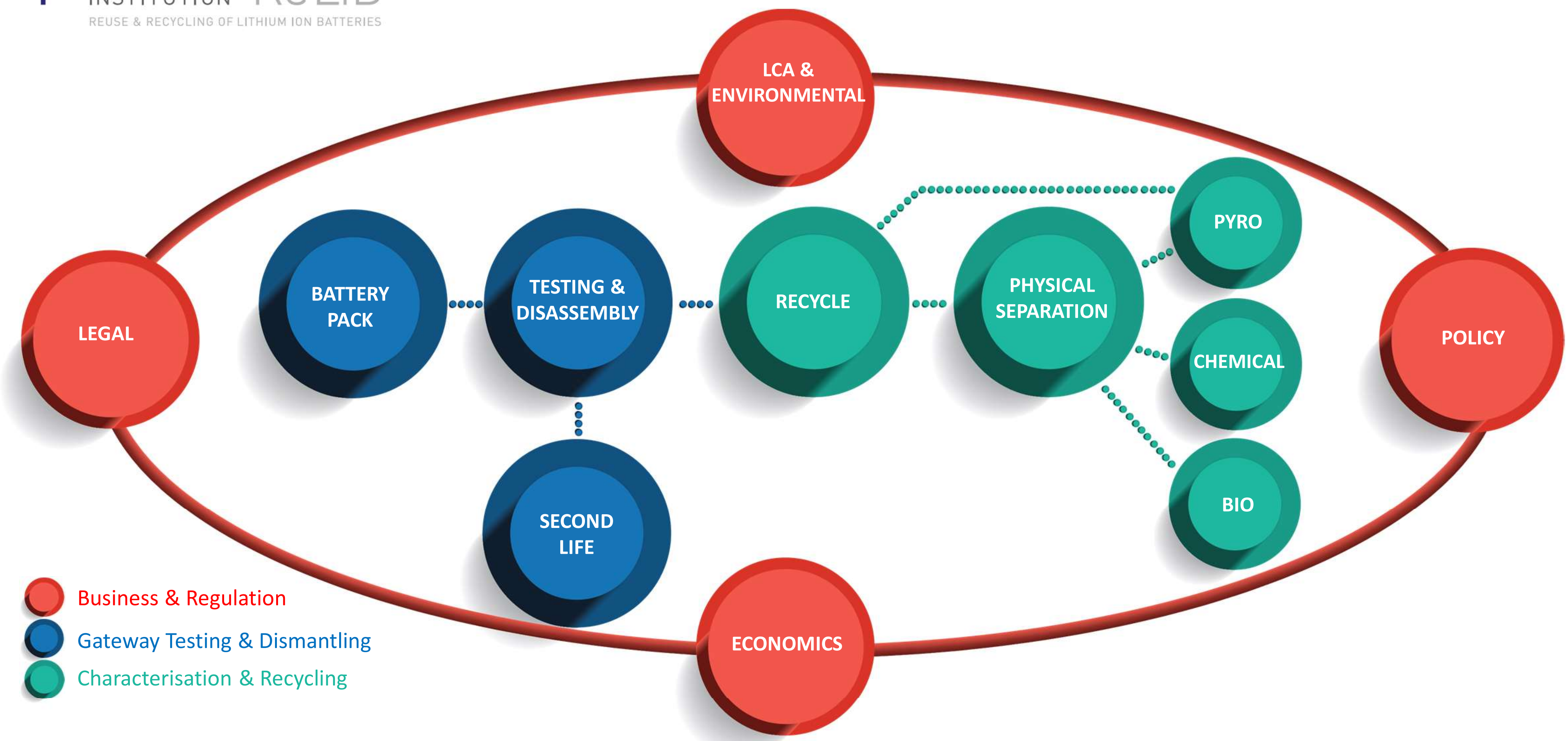
REUSE & RECYCLING OF LITHIUM ION BATTERIES

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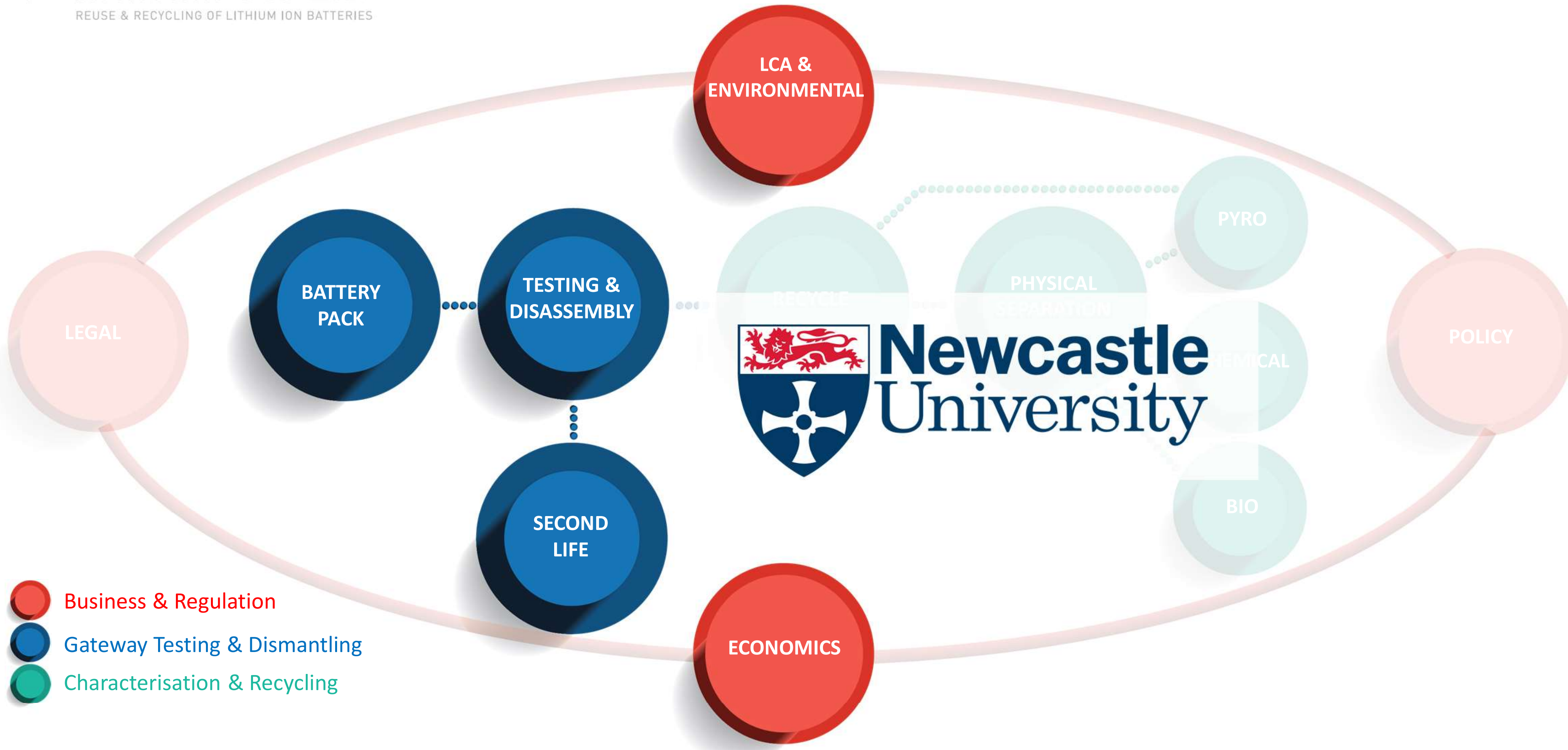


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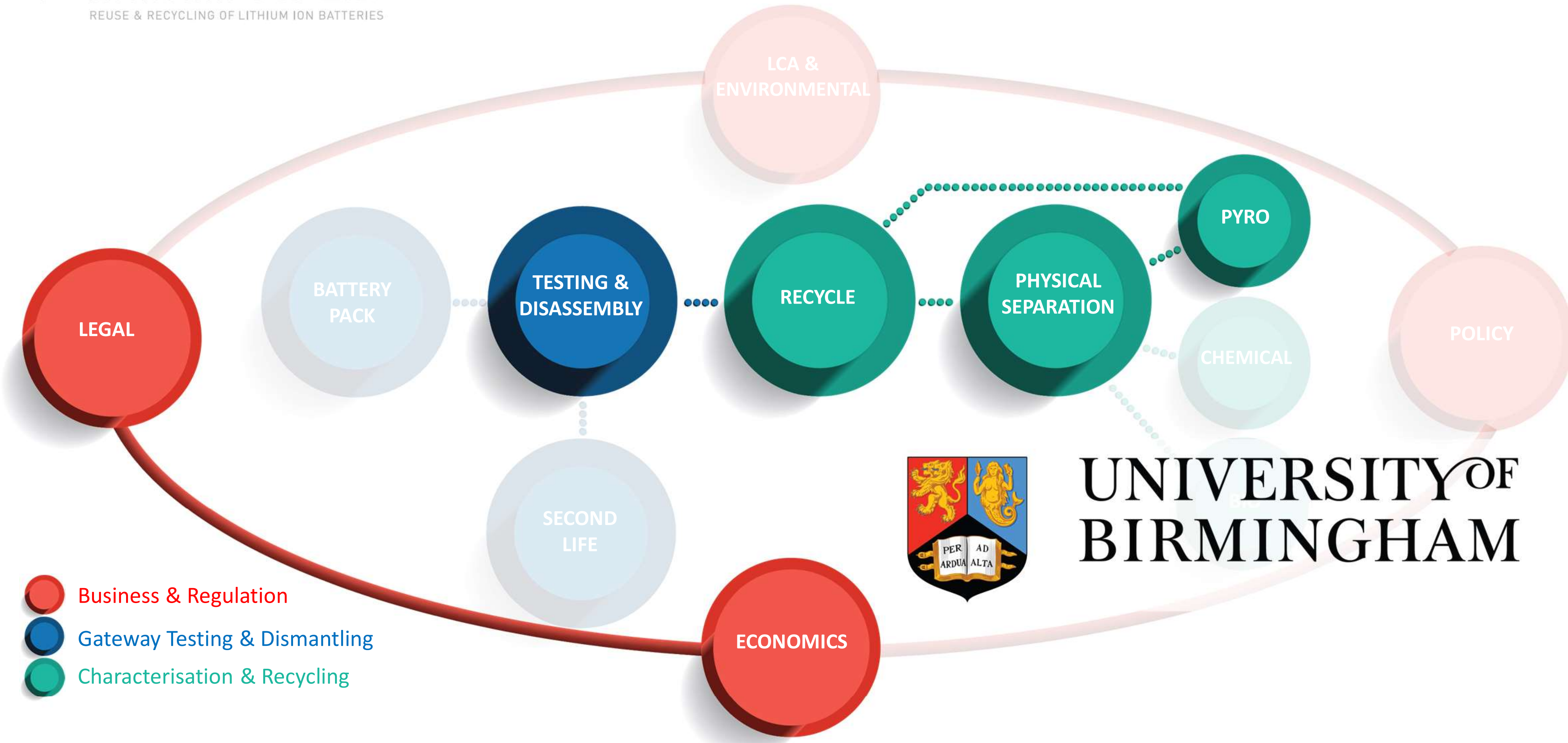


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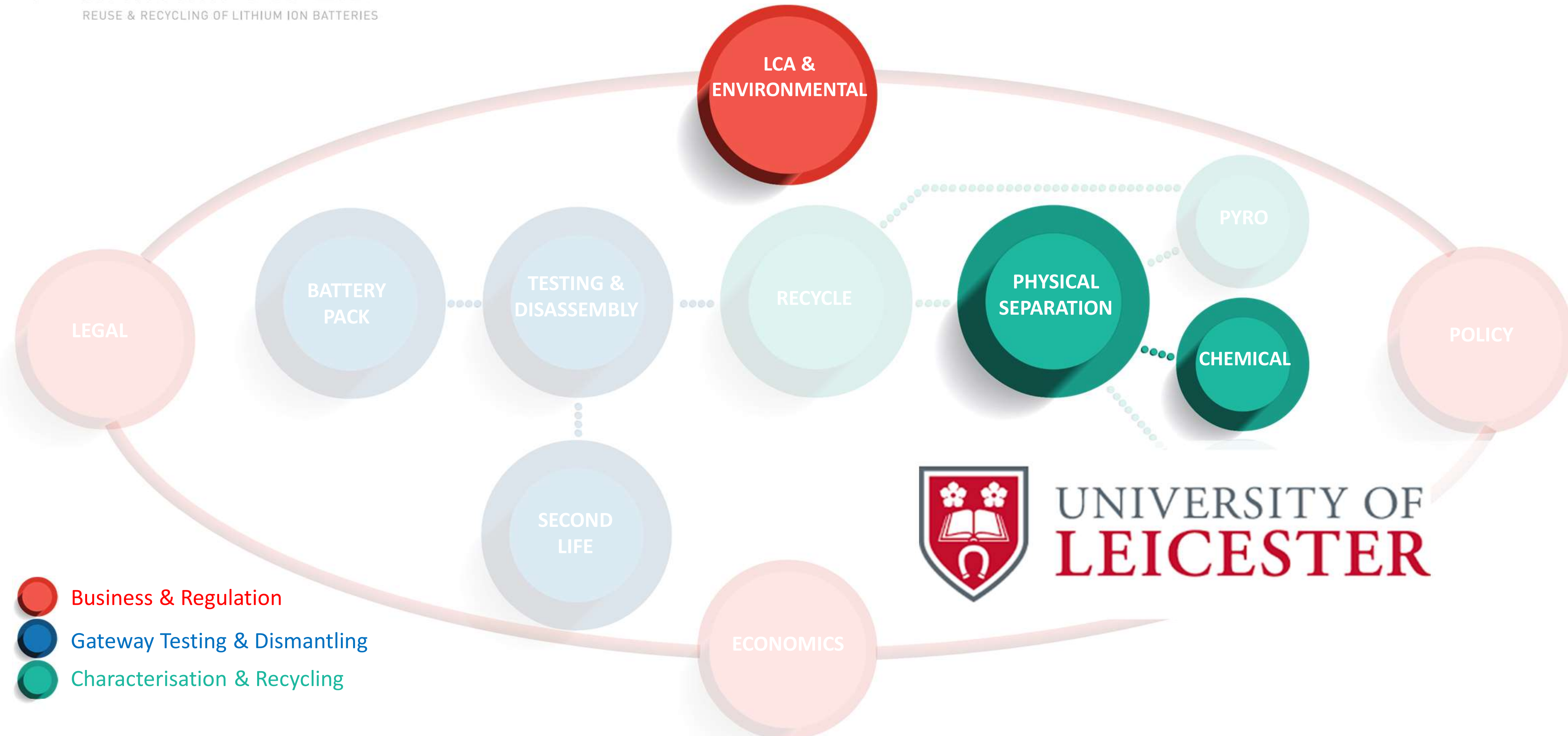
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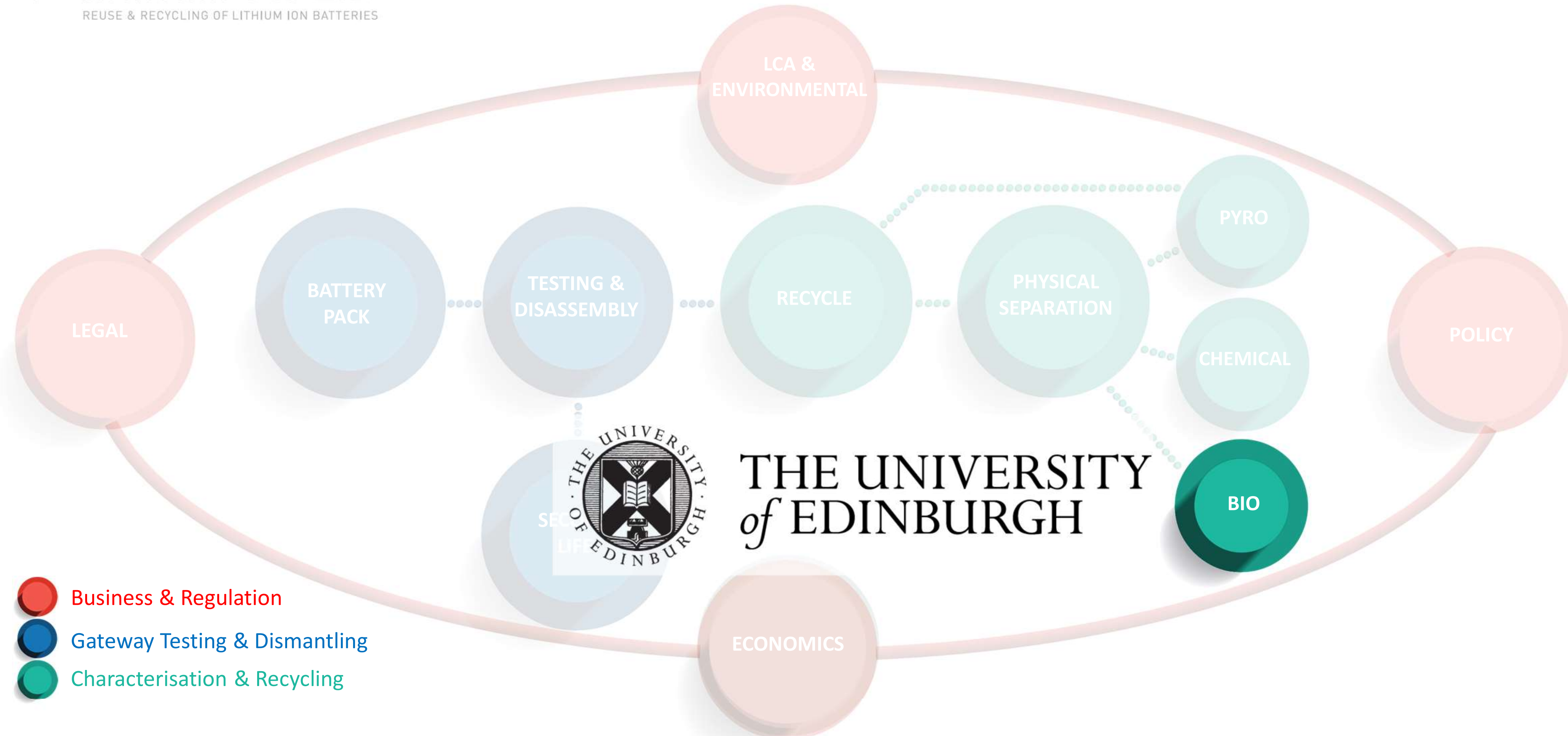


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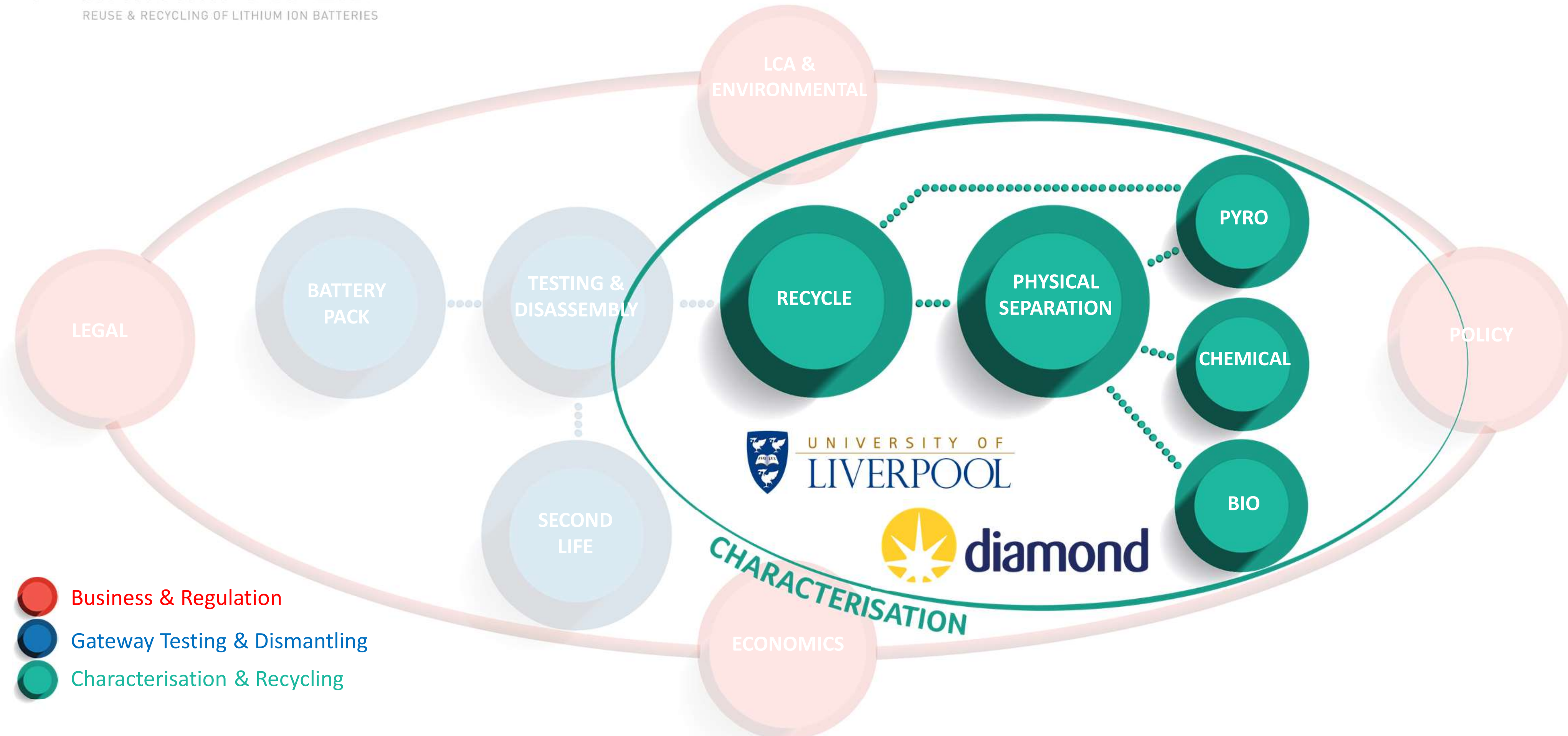

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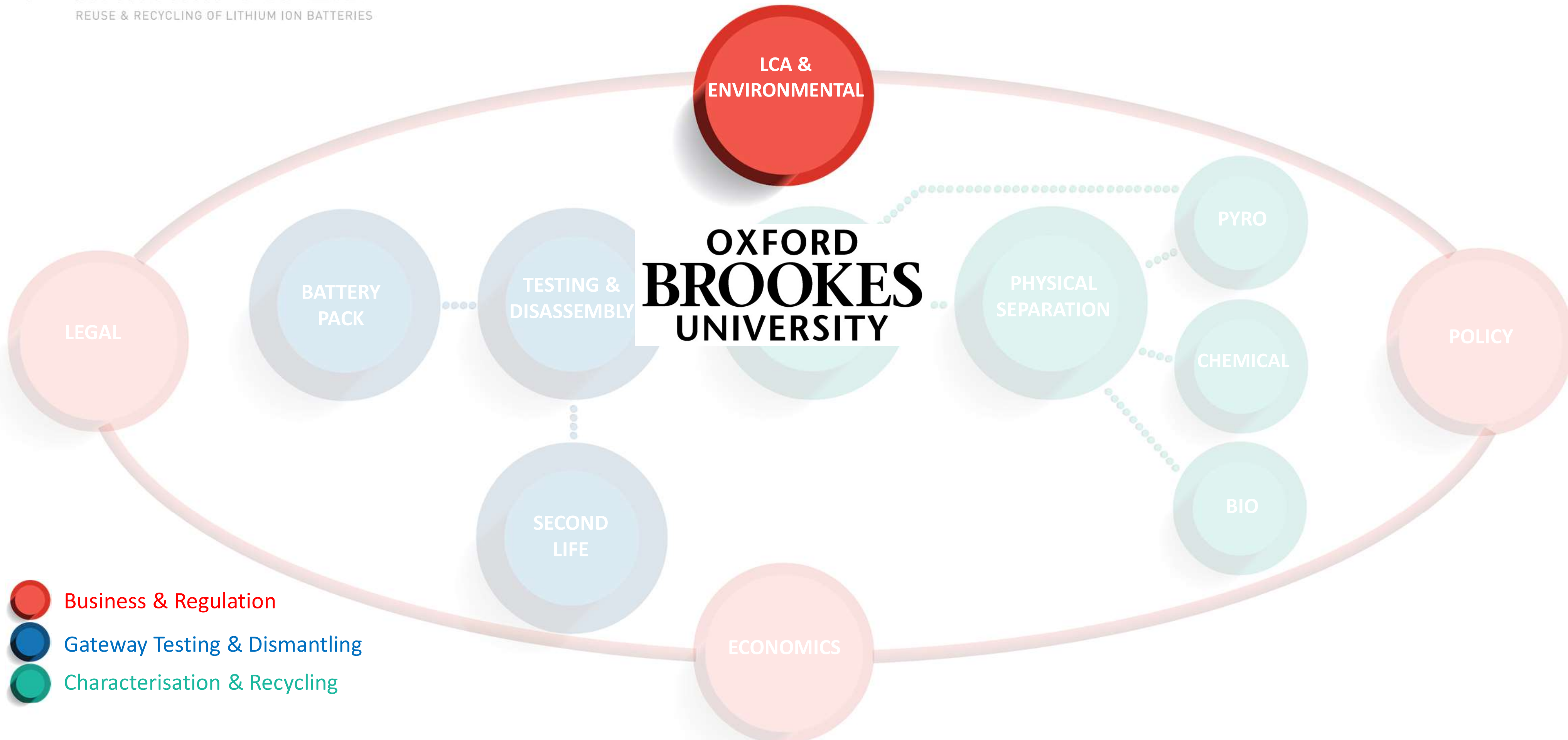

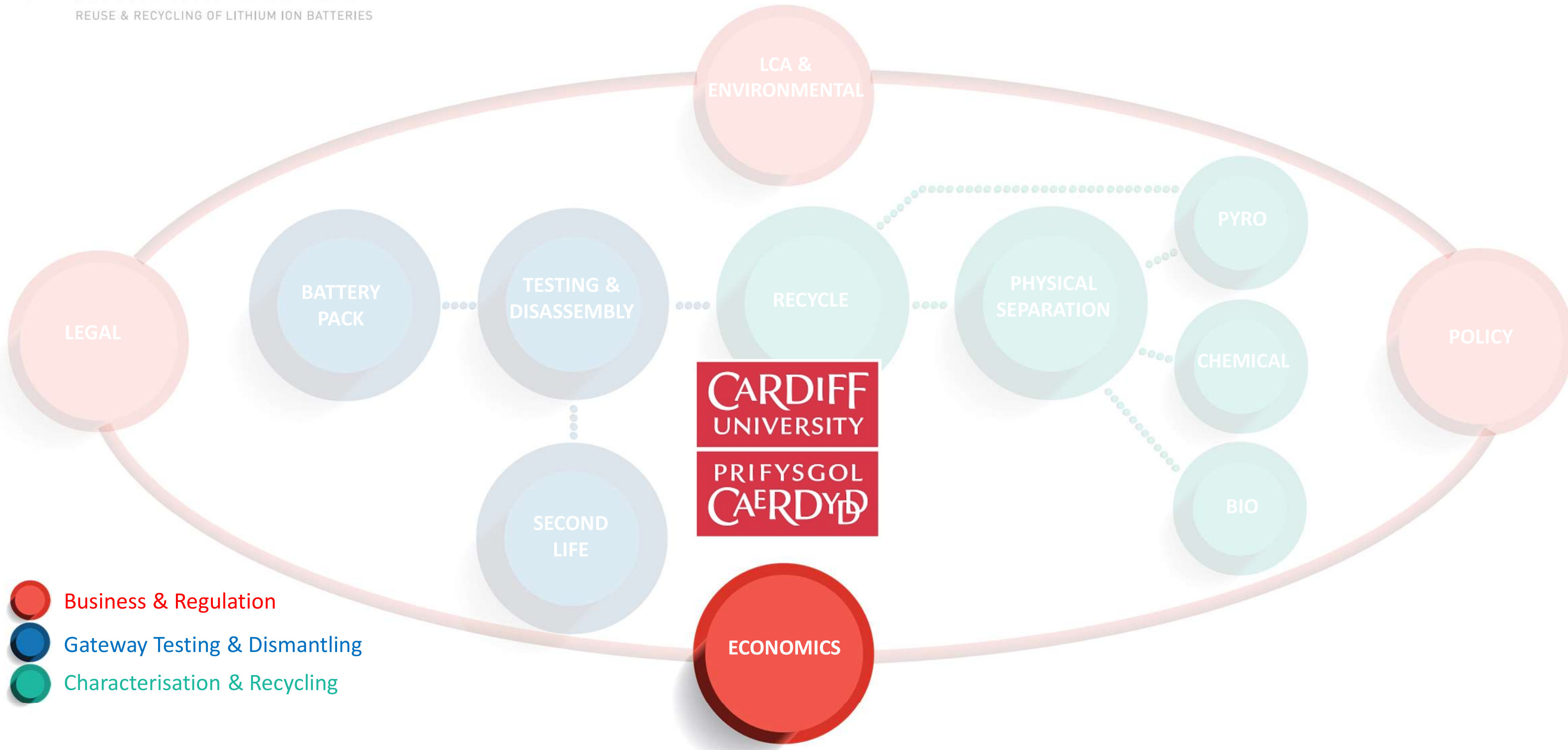
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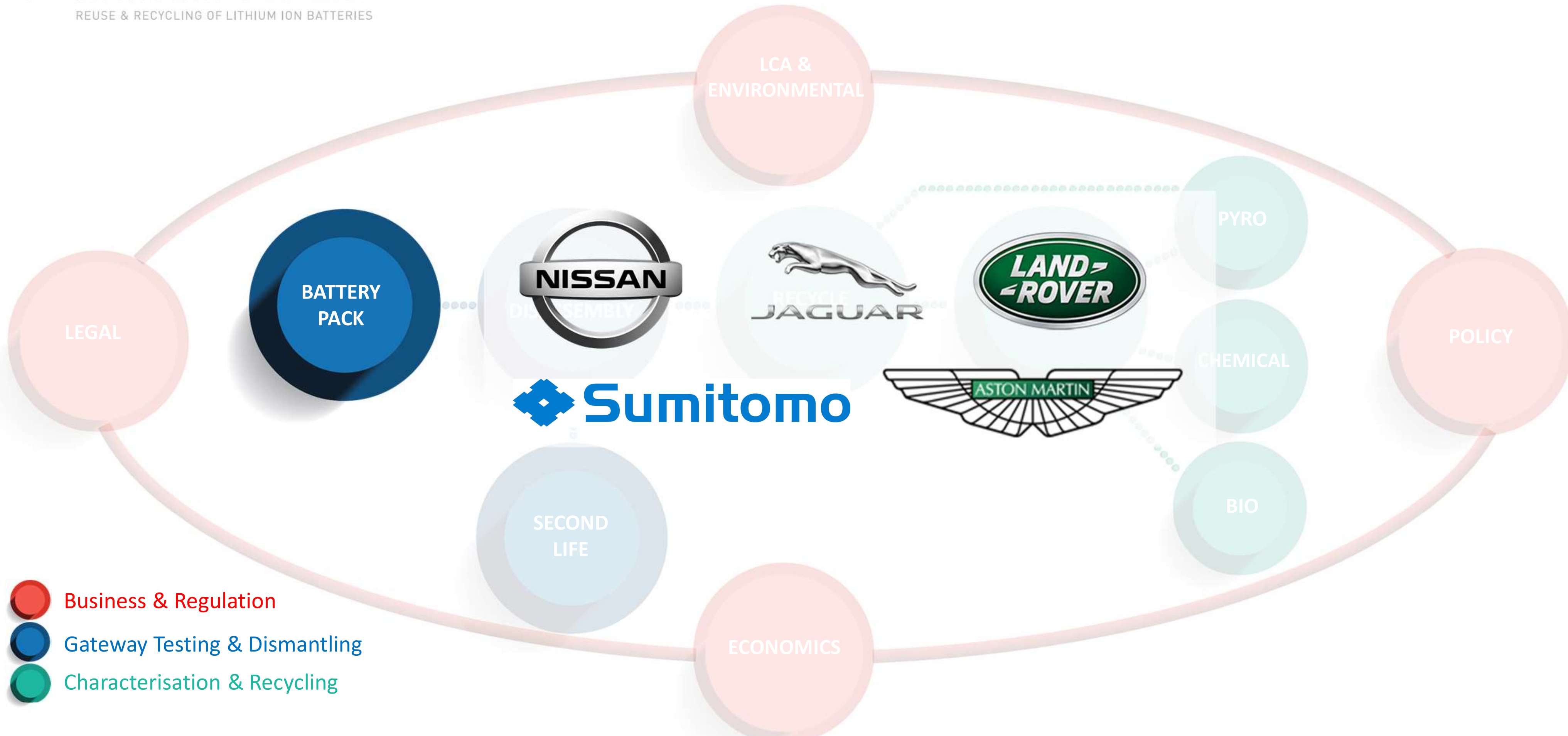


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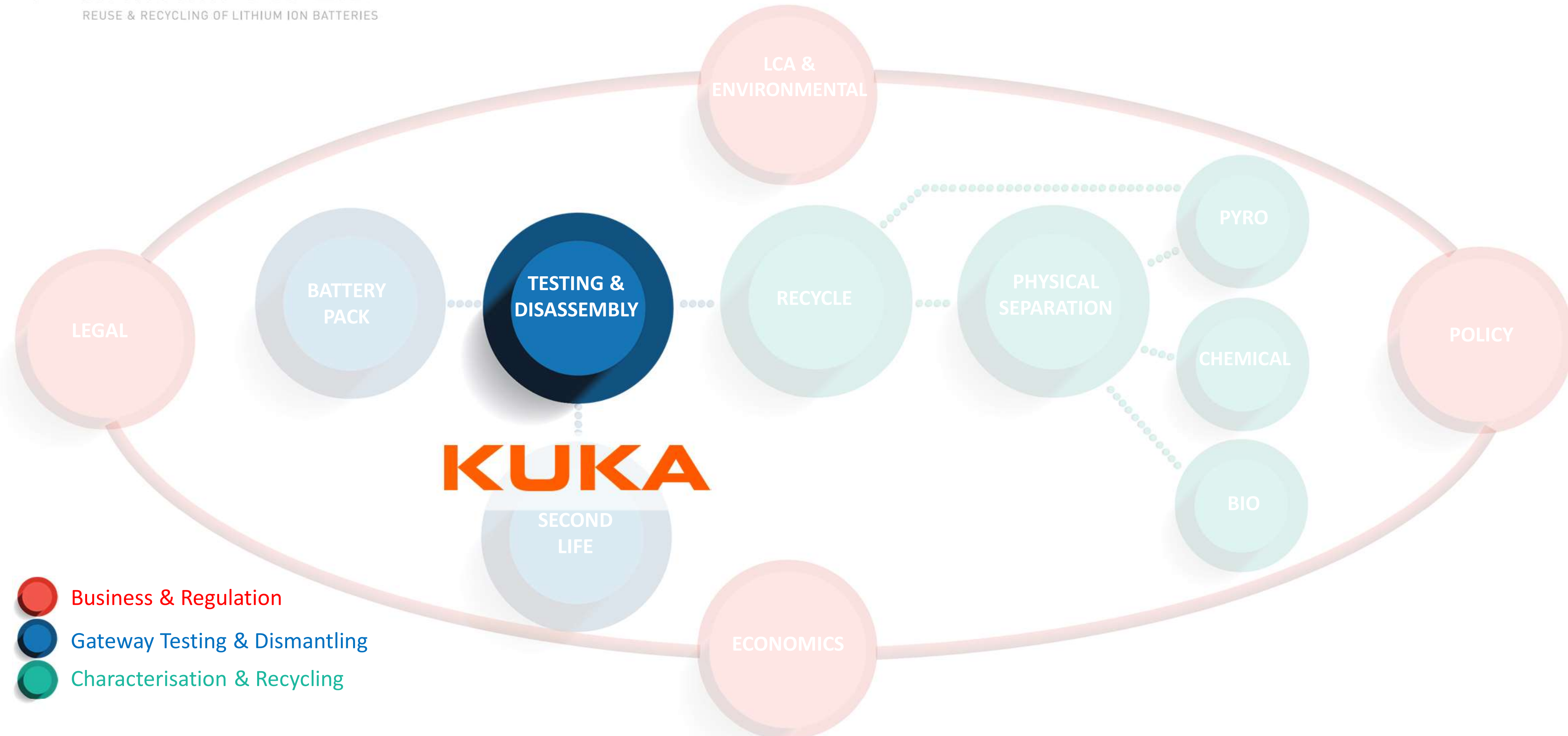


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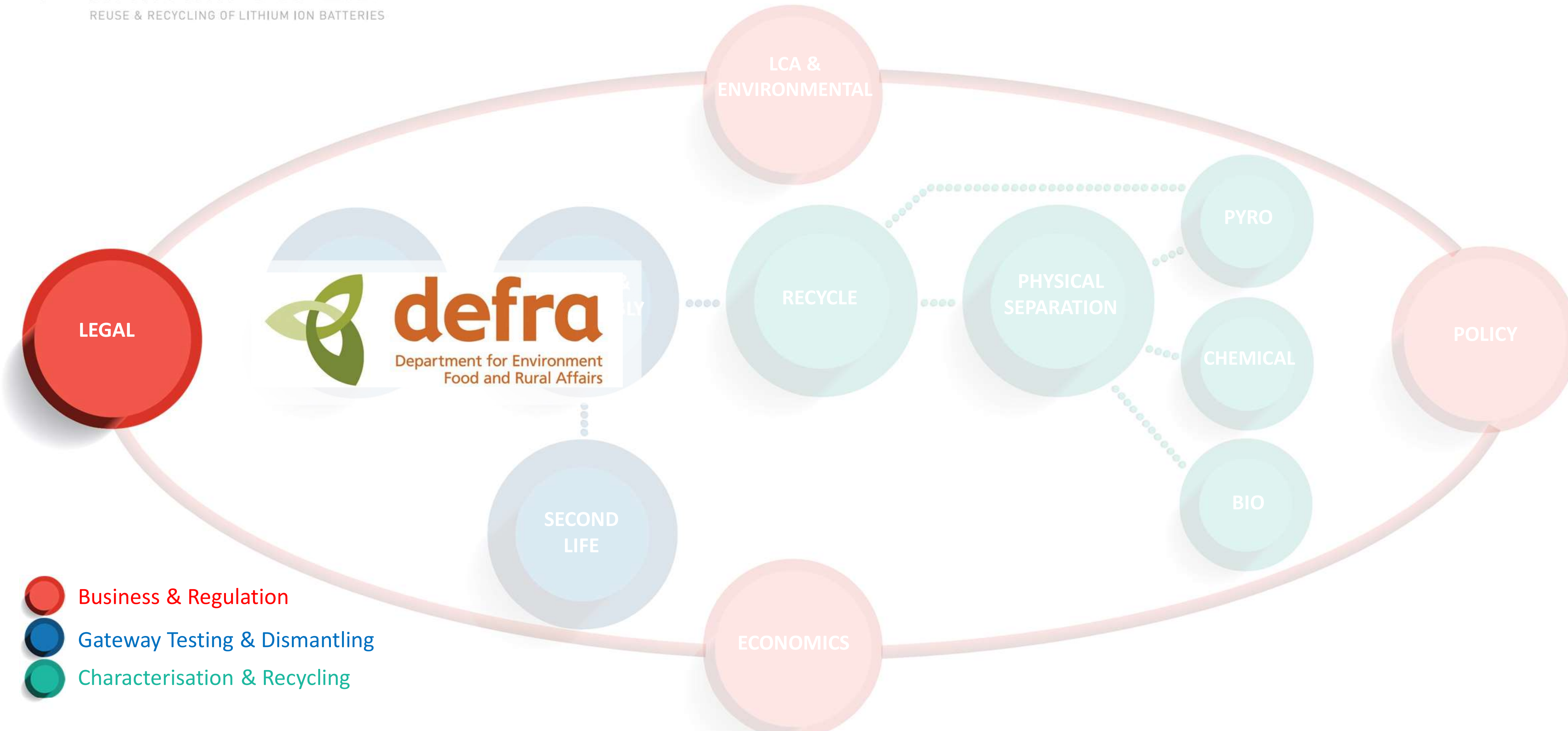

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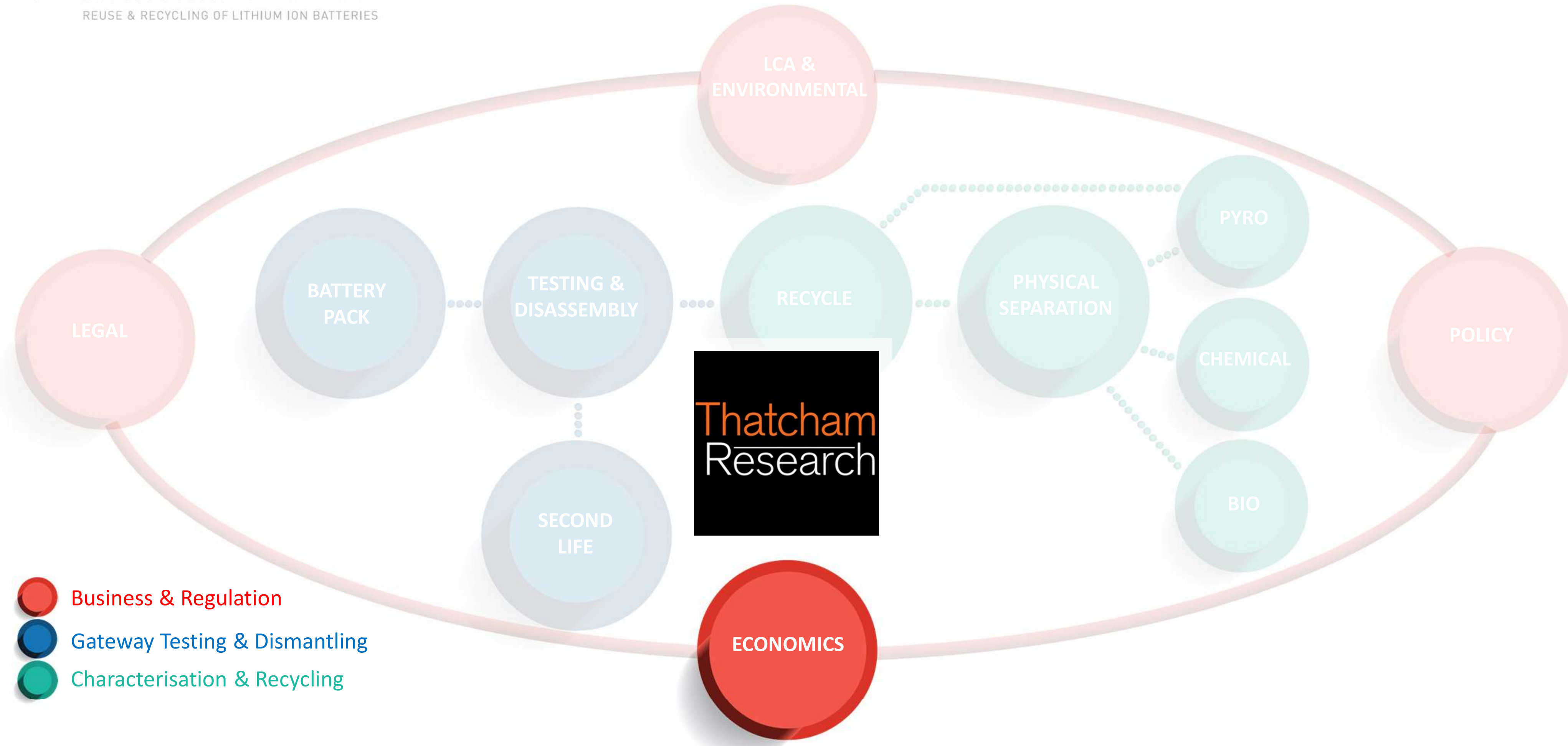


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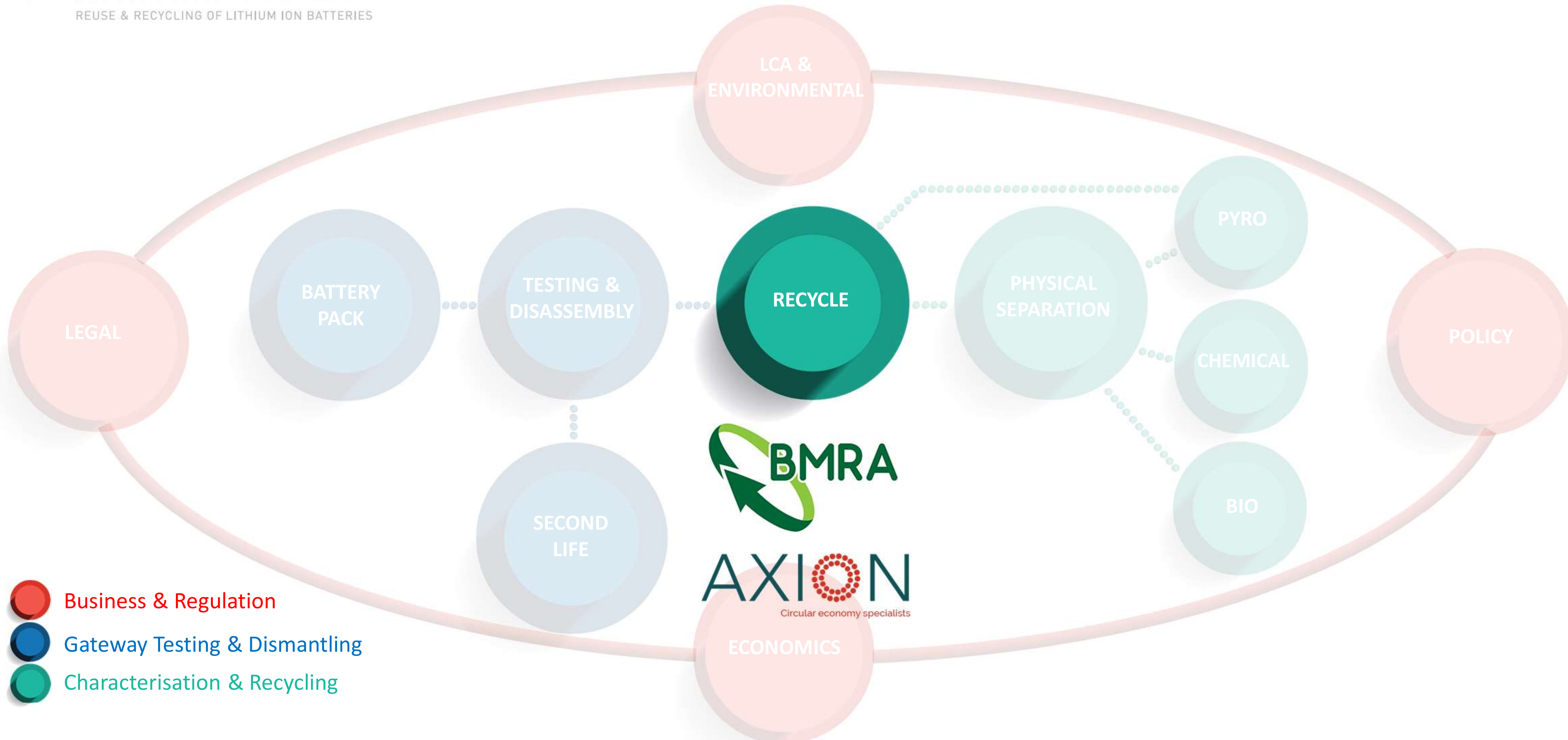


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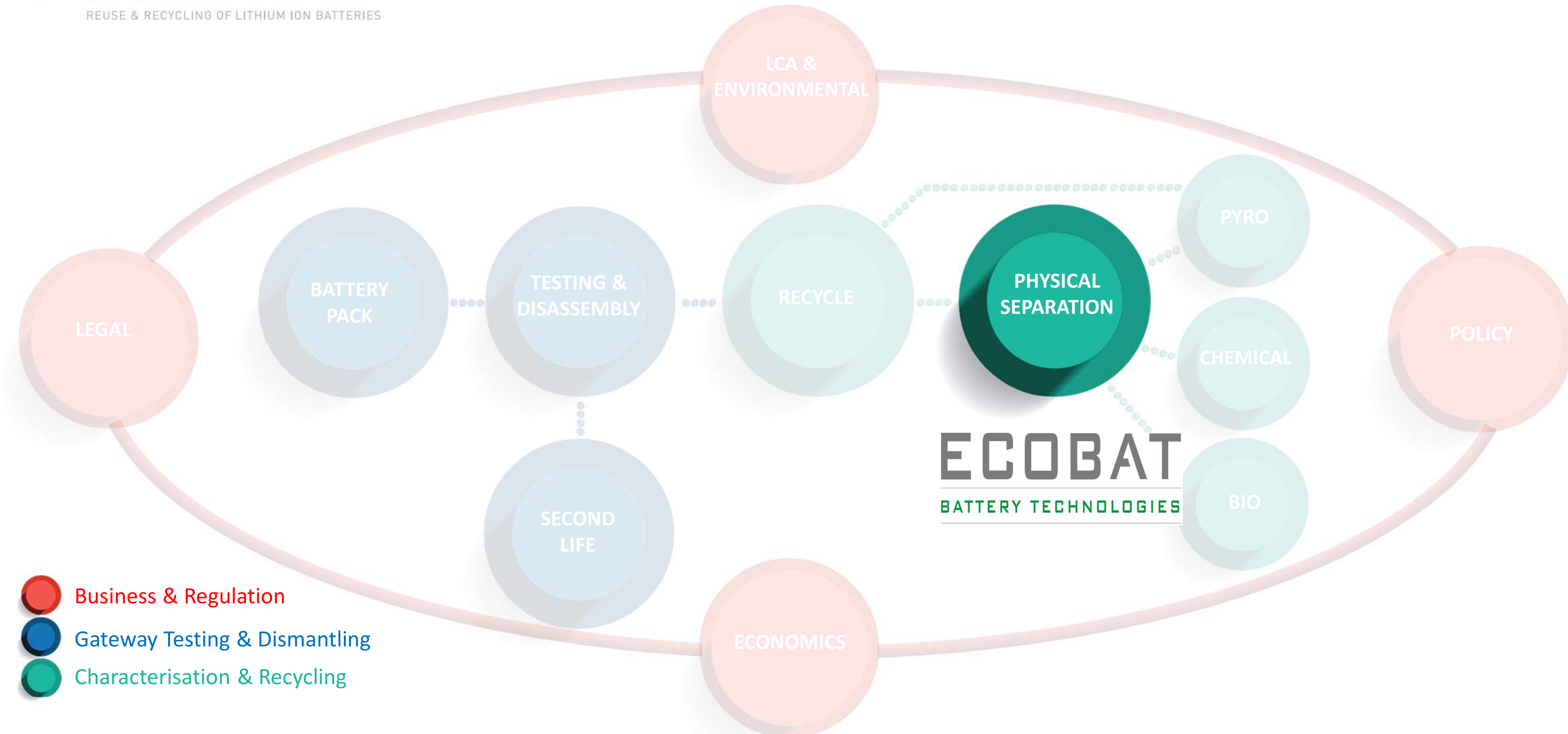


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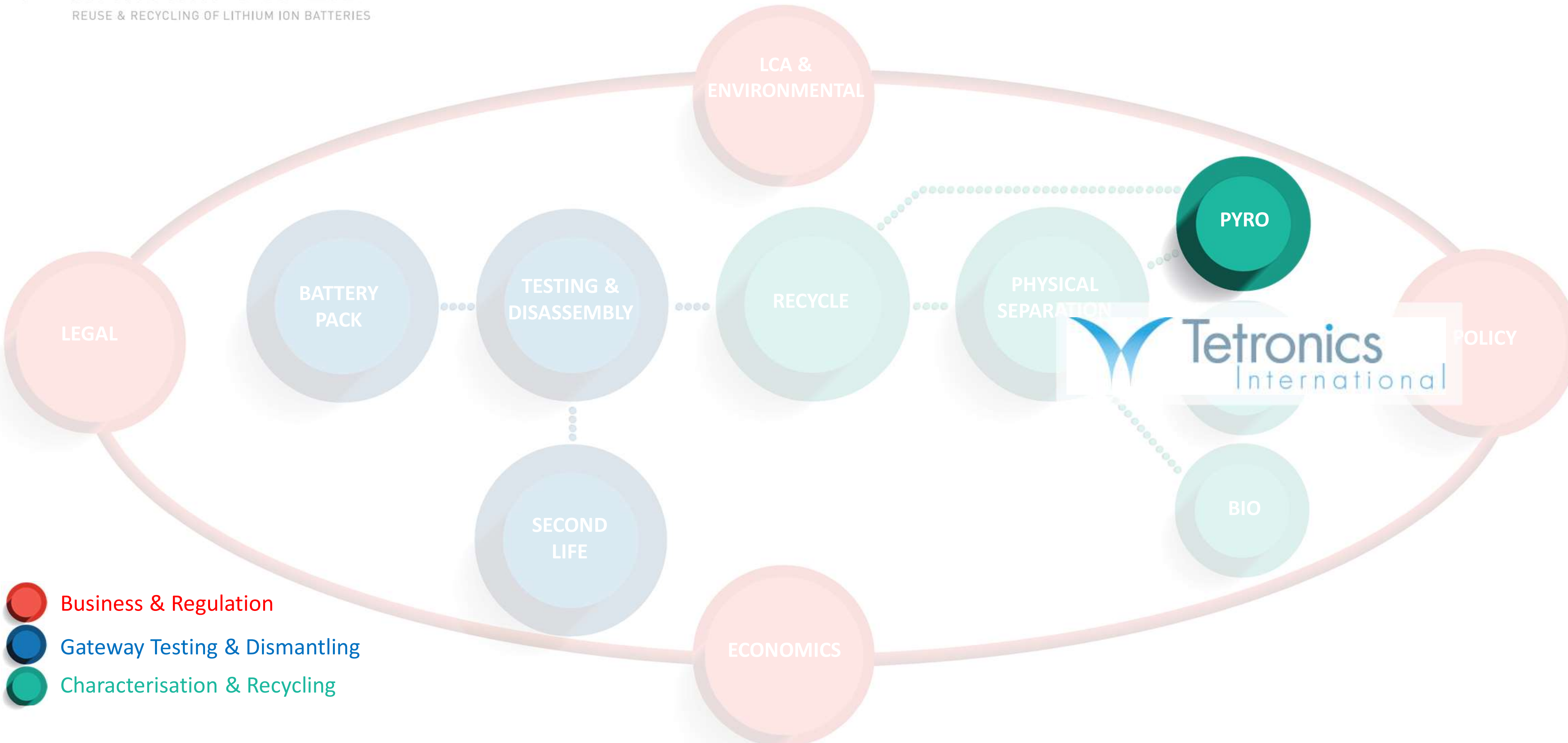


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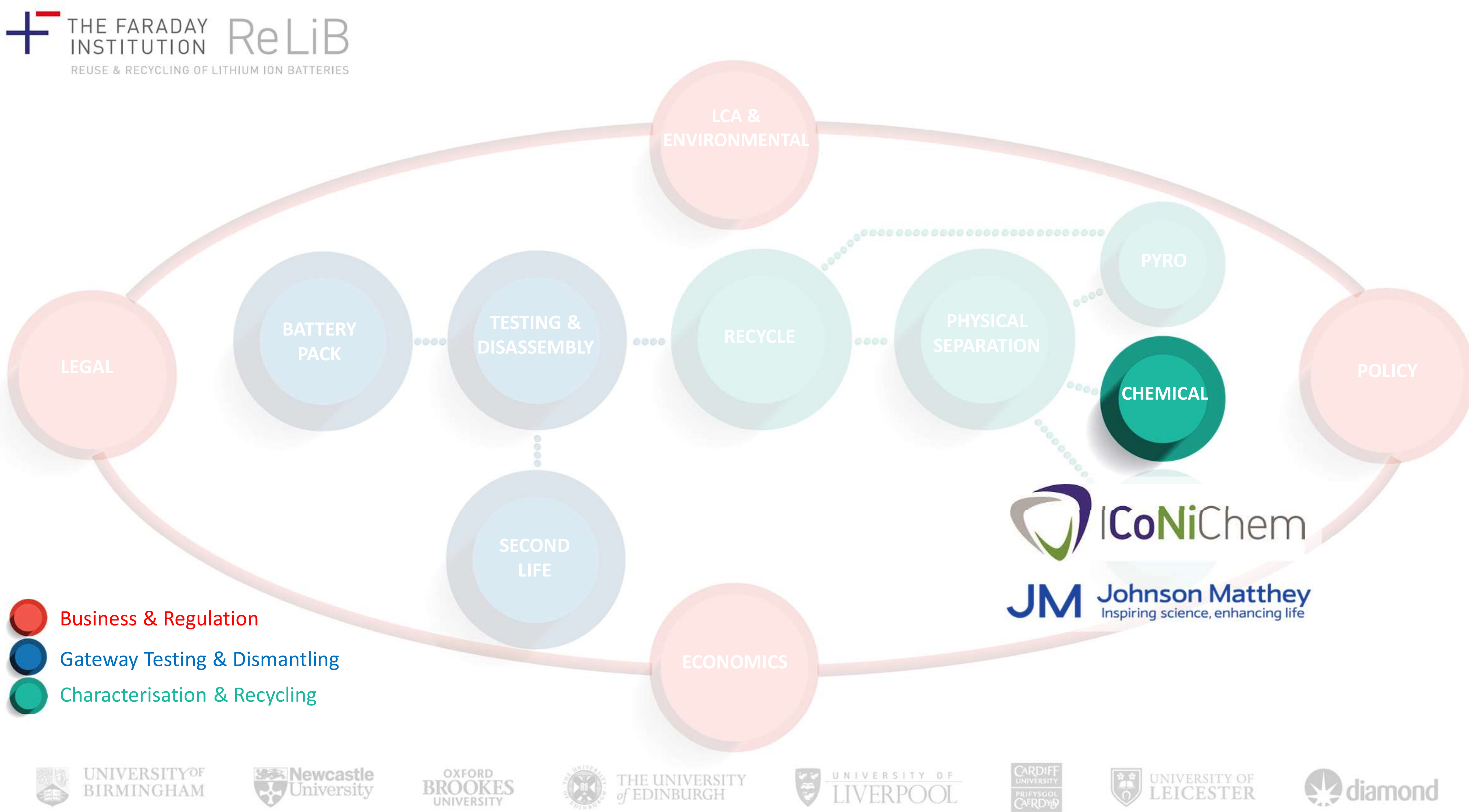
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# SAFETY

GATEWAY TESTING & DISMANTLING

Through our interactions with our industry partners, the emergency services and our own safety officers as well as in-depth literature review it has become evident that there is a deficit of knowledge, good practice and sharing of lessons learned in battery waste handling, transport and recycling sector in the UK.

## USED LEAF STORY

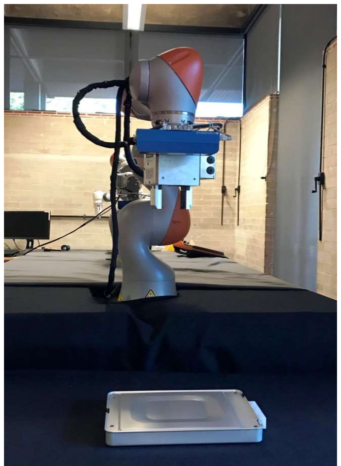
- Obtained 2011 Nissan Leaf
- 40,000 miles
- Removal of battery by partner (Sept 18)
- Ship to NU for disassembly, imaging and testing
- SIGNIFICANT SAFETY IMPLICATIONS – requirement to develop new stringent internal safety frameworks & assessments.


## HIGHLIGHTS

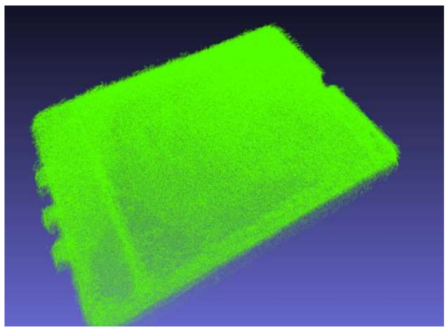
- Extensive modification of lab space, storage facilities generation of SOPs & RAs for dismantle & installation of safety equipment
- Consulted on safety by British Metals Recycling Association, MOD supplier, OEMs…
- Initial collaboration with T&W fire brigade
- Safety training for FI (Oxford review)- Dec 2018
- Development of safety training framework (All ReLiB researchers – 1st Session scheduled in January)

# ROBOTIC SENSING

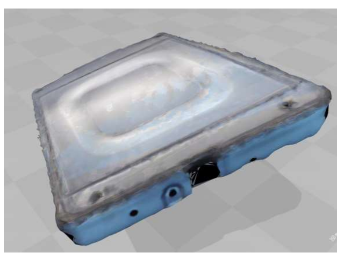
GATEWAY TESTING & DISMANTLING

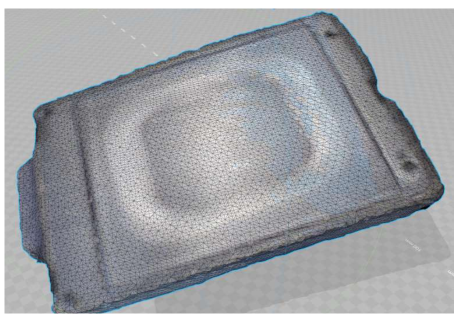


Module for scanning



Raw point-cloud images scanned from multiple views





Mesh model combining views

- Scanning and point cloud images have been generated for battery components (modules, cells, bolts )
- Scans have already been used to direct robots for disassembly of mocked up bolts.
- Mock ups of battery modules have been made by 3-D printing
- Smaller scale robots for module handling have been purchased
- High resolution cameras have been purchased and delivered to scan full battery packs
- Design of a battery pack disassembly line is in progress. The ReLiB team made a recent visit to the battery manufacturing plant in Sunderland. The activities there are confidential but the scope for further collaboration is being explored.

# SPECIFICATION OF 2ND USE APPLICATION DATABASE



- Power range
- C-rate
- Throughput during service
- In service depth of discharge
- Reliance on efficiency
- Underlying delivery pattern
- Frequency of events
- Minimum 2nd service life
- Value proposition
- Financial benefit

- These will be added to if additional notable use cases are judged to be relevant.
- The application characteristics are needed to model second-life battery performance under a range of secondary ageing profiles. This allows the demands on a battery in second life to be modelled and simulated.

- Techno-Economic Study for Pilot Pyrometallurgical Plant in UK

    Current BAT but difficult to achieve high material recovery rates

- Materials Segregation

    Potential to recover higher proportion of materials

- Bio-based Processing

    Longer term potential to recover specific nano-particles

- Advanced Characterisation

    Characterisation challenges in the development of more efficient recycling processes

# PYROMETALLURGICAL FACILITIES

CHARACTERISATION & RECYCLING

- Several Pyrometallurgical facilities are currently processing EV batteries
- Umicore (Belgium) has developed an industrial pilot plant that has the capacity to treat 7,000 tonnes/year (they also have battery pack dismantling facilities in Germany)
- Other smelting companies process batteries as part of their feed materials (e.g)

    Glencore | Canada & Norway

    NickelHutte | Germany
- No suitable primary metallurgical facilities in the UK.
- At present all EoL EV batteries are exported from the UK for reprocessing
- Should the UK rely on exporting EoL EV Batteries?

# TETRONICS: PYROMETALLURGICAL STUDY



- Designs and build small scale systems for the pyrometallurgical treatment of wastes and residues (plants to treat 5,000 – 20,000 tonnes/year)
- Conducting techno-economic study of the economics of a bespoke pyrometallurgical facility for battery recycling in the UK.
- Using typical input data and thermodynamic modelling to determine yields
- Will prepare costing for a industrial pilot plant in the UK.
- Techno Economic Assessment mid November 2018

# PHYSICAL SEPARATION - SCALE-UP - ECOBAT



Battery Shredder with $CO_2$ blanket at Ecobat

Density, size and Magnetic separation under $CO_2$

Shredded Battery Foils

Black Mass Separated Plastics

Modifications:

- Enlarged Air lock to accommodate A4 pouch cells.
- Screw conveyor from shredder to physical separation circuit for low density materials.
- Comminution control:
  - Additional modification of shredding teeth
  - Blanking plates to vary from shredding/ grinding axis
  - Upgrade of PLC (Programmable Logic Controller) for speed control
- Addition of a coarse shredder and blanketed conveyor to main shredding chamber for modules.

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# WHAT ARE THE BARRIERS TO MATERIAL SEGREGATION?

CHARACTERISATION & RECYCLING

**PVDF binder** is strong and homogeneously distributed- difficult to target solvents

**Approach**- better characterisation of materials and targeted choice of solvent systems

## 8 MONTH RESULTS

- Started material characterisation
- Scoped out useful solvents
- Trialed mechanical, physical and chemical separation of components. All function with different efficiencies.

## BARRIERS TO SUCCESS

- Late start of staff, but all now in place so progress should be faster in Q3 and 4
- Sample distribution – Now been achieved

## POSSIBLE ACCELERATORS

- Better picture of composition (Experiments planned with Diamond)
- More in depth mechanical testing – experiments planned at Leicester
- Added UG support to help with sample turnover

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# SOLVENT-BASED APPROACH

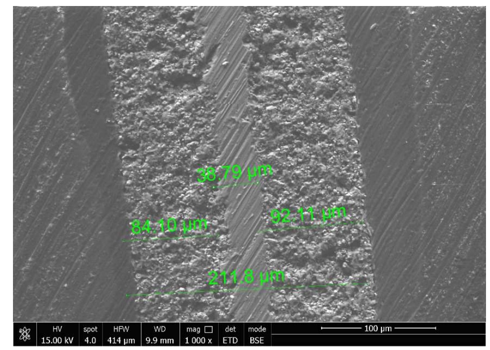


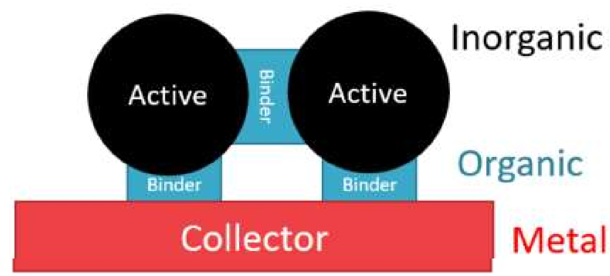
- For a solvent-based method to function the liquid must modify the 3-component phase boundary
- Critical questions include;

  -how to wet the surface?

  -is it better to dissolve or oxidise?

| | Solvent | |
|---|---|---|
| Binder | Organic solvent | Potentially best LCA<br>Intermediate cost, flammability, toxicity?<br>Design new solvent systems |
| Collectors | Oxidising agents | Aq. Acids –low cost – poor LCA<br>Ils – higher cost – better LCA? |
| Metal oxide | Acidic solutions | How is the active re-formulated? |
| Carbon | None | Residue – How is it reactivated? |





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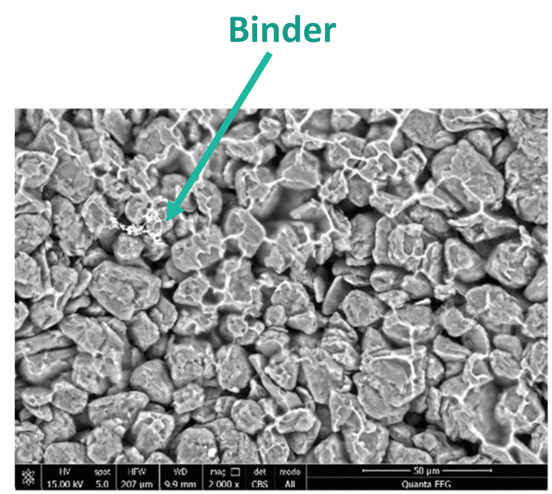
# SOLVENT-BASED APPROACH- PRELIMINARY RESULTS

CHARACTERISATION & RECYCLING

- How can we break the 3 component interface to aid component segregation ?
- Where is the binder?
- What does the metal-binder interface look like?
- How can we promote binder solvation?
- Can we design the solvent system with the best LCA/ process economics?

| | Solvent | | Time scale |
|---|---|---|---|
| DMF | Organic solvent | Releases particles but slow to penetrate matrix* | 1 day – 1h with ultrasound! |
| HCl, $H_2SO_4$<br>$I_2$ in DES | Oxidising agents | Aq. Acids – Digest both Al and Cu<br>DESs – slower than acids but electrocatalytic | 1-2 h<br>1 day |
| Metal oxide | Acidic solutions | Dissolve metal oxide but how is the active cheaply re-formulated with a good LCA? | 1 day |
| Carbon | None | Residue – How is it reactivated? | |

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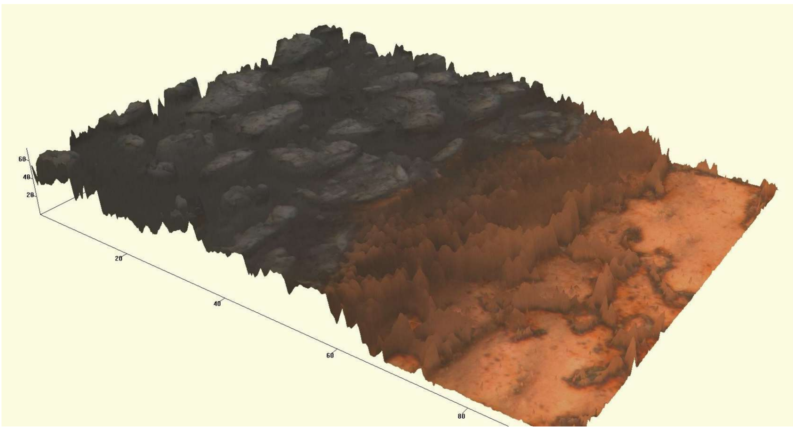
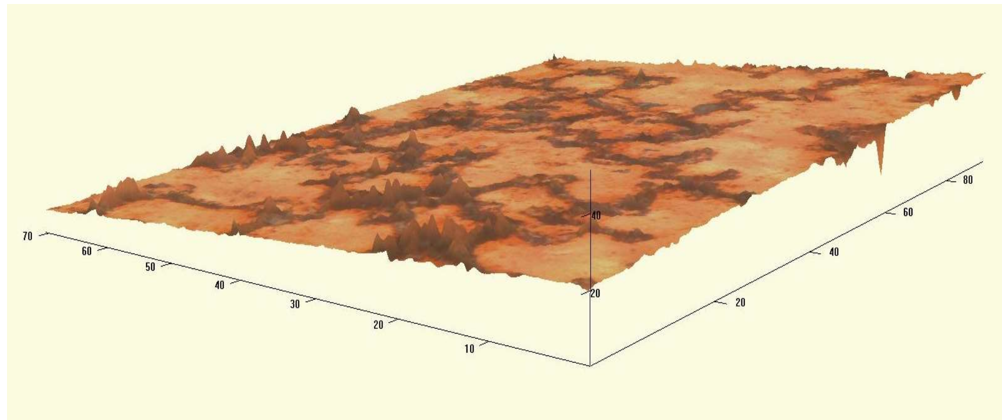
# PROGRESS



- Able to break C-Cu interface in a benign and fast method (<5 s)
- Need to adapt it to function on Al-MO interface



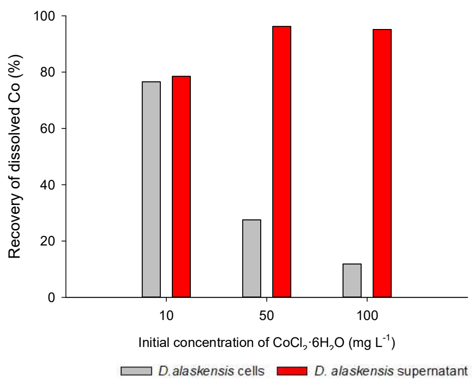


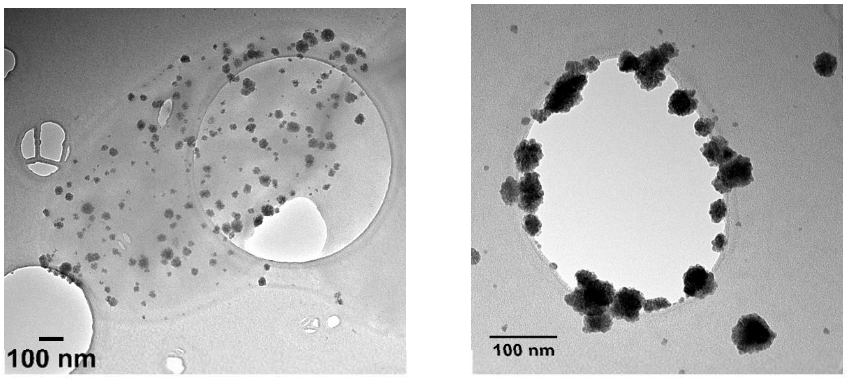
# BIOPROCESSING



## Bacterial specificity for metals

- Ni and Co
  - *D. alaskensis* can precipitate Co and Ni ions
  - High recovery (aprox. 100%) of dissolved $Co^{2+}$ at concentrations between 10 and 100mg $L^{-1}$.
  - Co-based nanoparticles have been produced and delivered to UoLi for characterisation
  - Currently we are investigating the recovery of Co and Ni in mixed metal samples. Nanoparticles produced in presence of one or two metals (Ni and Co) have been delivered to UoLi for characterisation.




TEM image of Co-based nanoparticles attached to the outer membrane of *D. alaskensis* at 10 mg $L^{-1}$ (left) and free nanoparticles produced at 1 mM (59 mg $L^{-1}$ )(right).

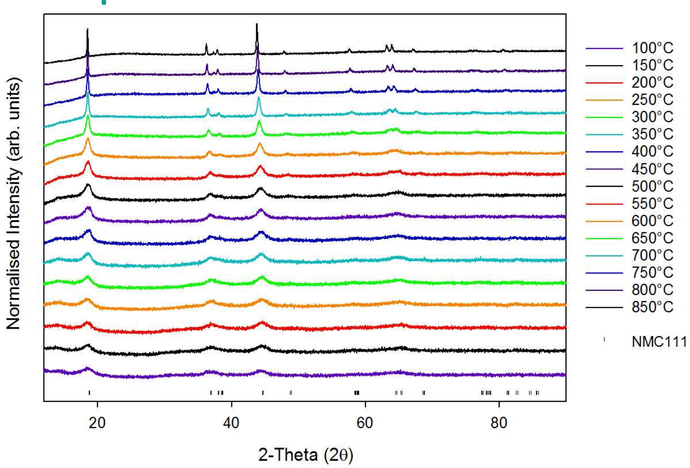
# SHORT LOOP RECYCLING



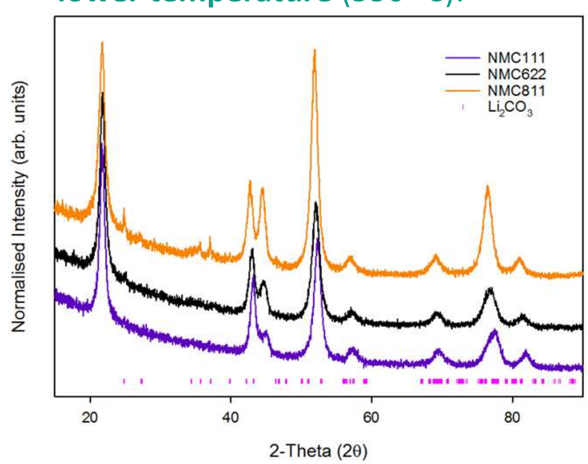
$$\text{Sol}^{n}\text{ of Ni, Mn, Co} \longrightarrow \underbrace{Ni_xMn_yCo_z(OH)_2}_{\text{Composition controlled via solution}} \xrightarrow[850\ ^{o}C]{Li_2CO_3} LiNi_xMn_yCo_zO_2$$ [1]

Mild oxidation + mechanochemical synthesis $\longrightarrow LiNi_xMn_yCo_zO_2$

Preliminary studies indicate that NMC is formed at **room temperature**




Variable temperature studies shows that NMC forms at significantly **lower temperature** (550 $^{o}$C):




**Next steps:**

- Electrochemistry and local structure as $f^n$ of T (X-ray/neutron proposal submitted)
- Recycle cells at various states-of-health

[1] R. Zheng, W. Wang, Y. Dai, Q. Ma, Y. Liu, D. Mu, R. Li, J. Ren, C. Dai, A closed-loop process for recycling $LiNi_xCo_yMn_{(1-x-y)}O_2$ from mixed cathode materials of lithium-ion batteries, Green Energy Environ. 2 (2017)

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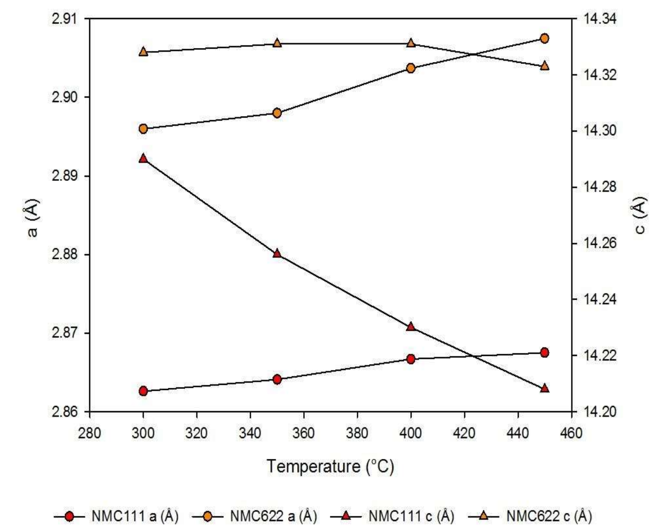
# DIRECT RECYCLING OF CATHODE MATERIAL

CHARACTERISATION & RECYCLING

- Fluorine is present in the electrolyte (+SEI) and in PVDF binders
- General assumption is that binder is unreactive and will simply burn off
- However, PVDF has been shown to act as a fluorination reagent at relatively low temperatures (350 °C) [1
- Evidence for variation in cell parameters with heat treatment (compositional changes)
- **Future work:** investigating chemical composition of electrode materials at various states-of-health, before and after separation from the current collector. How does vary spatially within the cell and with different particle morphologies?
- What is the effect of impurities on the electrochemistry of the recycled material?




*Cell parameters of NMC111 and NMC622 after 12 hour heat treatments in air at different temperatures.*

[1] Slater P. R. and co-workers, Journal of Solid State Chemistry (2002) 186, 195-203

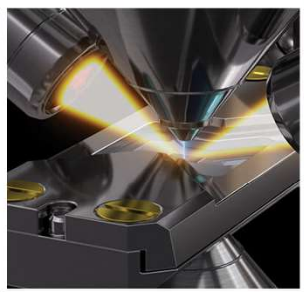
# PROGRESS TO DATE



## In-situ Scanning Transmission Electron Microscopy Characterisation of Pristine, EoL & Recycled Materials & Processes

- Established in-situ & low-dose methods for beam sensitive battery samples and chemical & biological recycling environments: can now test composition/structure and new protocols for the project
- Use microscopy methods to control different parameters (temperature, chemical composition, surface area/facets, solvent type and concentration, biomolecular species etc…



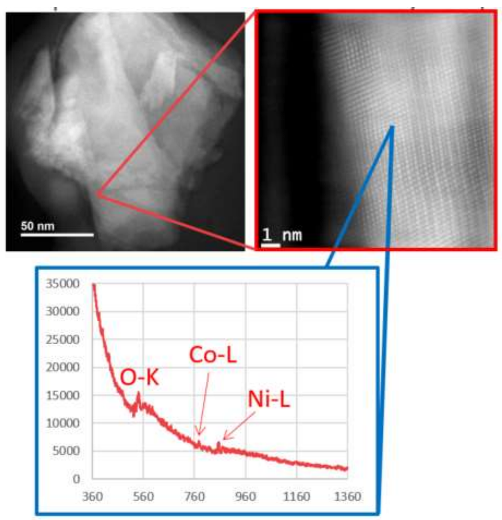


# PROGRESS TO DATE

CHARACTERISATION & RECYCLING

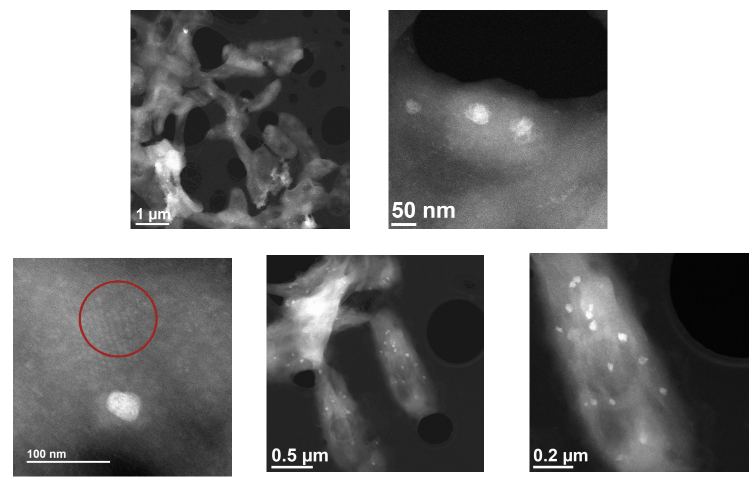
Initial experiments underway with samples from Leicester (chemical routes) & Edinburgh (biological routes)

- Example: Atomic scale distribution of elements in pristine and end of life battery components




Initial results show local structure variations and elemental distributions – synergy with degradation project

- Example: key kinetic factors controlling recycling processes via biological routes




Preliminary work shows distribution Co-based nanoparticle in bacteria cells

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# MATERIALS CHARACTERISATION AT DIAMOND

CHARACTERISATION & RECYCLING

- How and why do pristine and recycled LIB materials from various recycling routes differ?
  - For the successful use of recycled materials we must demonstrate that they are able to complete with pristine materials. Understanding any difference in performance will be vital to improve on the recycling methodology.
  - *Method: Ex-situ* characterisation of recycled materials and *in-situ* studies on cells prepared from these materials will inform any differences between pristine and recycled materials.
- Can we better correlate EIS with structural failure of battery packs at multiple length-scales?
  - Differentiating between types of cell failure and correlating this with non-destructive testing will better inform efficient recycling paths though more thorough cell-level triage.
  - *Method:* Measure cells from different points in their life-cycle with EIS, then fully characterise cells using a range of non-destructive and post-reclamation methods at multiple length-scales to correlate EIS behaviour with different failure paths.
- Can we understand and tune the processes occurring during reclamation?
  - Understanding the fundamental mechanisms in the chemical reprocessing steps in real-time will lead to more efficient recycling routes.
  - *Method: Perform in-situ structural characterisation of materials during processes such as solvent reclamation*.

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# PROGRESS TO DATE

BUSINESS & REGULATION

- Literature review on circular economy approaches and extended producer responsibility
- Establishment of legal advice and assistance programme
- Work with Defra/EA on reform of batteries regulation

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# CURRENT ISSUES



- ELV recycling rates
- Circular economy package
  - Article 8a Waste Framework Directive
- Batteries review
- Resources & waste strategy
  - Review of EPR schemes including batteries
  - BEIS: Office of Product Safety and Standards: producer compliance review
- Brexit
  - Netting off

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# WORK UNDERWAY

BUSINESS & REGULATION

- A textual explanation of the regulatory framework for lithium-ion batteries across sectors
- A gap analysis of the regulation of lithium-ion batteries which maps onto the life cycle of the battery
- A review of extended producer responsibility schemes and circular economy approaches in areas such as waste electronics, end of life vehicles, waste packaging and other areas that might inform problems of battery waste

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## *AUTOMOTIVE*

| Life-cycle: | Research and Development/Design | Battery Manufacture/production | Vehicle assembly | Supply: 1. Wholesale | Placement on the Market | Use | Servicing of battery | End of Life/disposal |
|---|---|---|---|---|---|---|---|---|
| ***Product Liability/Consumer Protection*** | | | | | | | | |
| Consumer Rights Act 2015 | | X | X | X | X | X | X | Relates to s19 SOGA 'reservation of right of disposal' 1979 X |
| General Product Safety Regulations 2005 (GPSR 2005), SI 2005/1803 | X | X | X | X | X | X | ? | |
| Unfair Terms in Consumer Contracts Regulations 1999, SI 1999/2083 | | | | X | | X | X | |
| Sale and Supply of Goods Act 1994 | | Fault in the manufacturing process X | Fault apparent from assembly X | Bulk s2(2)(b) X | | X | X | |

# KEY CHALLENGES

- How can we design EV LiBs for recycle and manufacture at length scales from the molecular to battery pack level (ten orders of magnitude)?

- Can we characterise chemical distribution of elements, phase information and morphological changes in used EV cells in a spatially resolved manner to drive world-leading recycling processes (and battery science)?