MAKING AND TESTING CATHODE MATERIALS

Preparation and characterization of advanced coatings on cathode materials

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Abstract and Motivation

Characterization is a very important process in materials science. It involves determining a material's structure or properties by using many different analytical techniques. A small improvement during the research and development stage of a coating will lead to a massive improvement in performance and efficiency or a large decrease in costs when the coating is produced at a large scale.

Example of materials that could be used for the preparation of a cathode coating:

OX-1 Components (BASELINE)	Materials	Percentage (%)	Solid weight (g)	Solution Solid content (wt.%)	Solution weight (g)
Active	NMC 622	96.00	97.92	100.00	
Conductive additive	C65	2.00	2.04	100.00	
Binder	PVDF	2.00	2.04	8.00	25.50
Solvent	Extra NMP				24.54
	Total	100.00	102.00		50.04
				Solids weight (g)	102.00
				Total material weight (g)	150.00
				Final Slurry solids (wt.%)	68.00

Preparation

- The primary step to making a cathode coating is to mix all the required components. First the active material (NMC/LFP) and carbon black are mixed using a Thinky Mixer (a) at 1300 RMP, to make them as homogenous as possible before any solutions are added. Then binder is added, for which I used 8% PVDF in NMP. The last step of the mixing process involves adding the solvent, for example, NMP. This is to decrease the solid content of the slurry, ensuring all powders are dissolved, and to reach a suitable viscosity to be used when coating. [1]
- To evenly spread the slurry onto a current collector (Al foil) I used an Erichsen Coatmaster 510 (b). The surface of the coater has a vacuum to ensure that your chosen current collector is flat and the gap between your applicator (c) and the foil is consistent. This model of coater allows you to set your coating speed (4mm/s). The other variable to consider is the applicator gap. A larger gap allows more slurry through and gives a higher coatweight.
- Finally, the coating should be heated at 80°C on a hotplate (d) until it is visibly dry, and then placed in a vacuum oven at 120°C for storage and to ensure there is no solvent remaining in the coating.

a

b

c

d

Slurry Characterization

- Rheology tells you the viscosity of a sample and how it is affected by differing shear rates. The slurries that I worked with during this internship were shear thinning, which means as the shear rate increases, the viscosity of the sample will decrease. For a slurry to be used in higher throughput, it needs to have a viscosity between 2 and 5Pa·s, at a shear-rate of $10s^{-1}$. I used the TA HR20 rheometer (e) with the parallel plate geometry.
- Hegman gauges (f) are used to see if any large/ coarse particles or agglomerates are in the sample. I recorded D_{100}/D_{50} values to show both the size and abundance of the largest particles. Large particle sizes will cause issues when the slurry is coated. The slurries I was working with had D_{100}/D_{50} values of 20/15µm.
- Solid content is an important value to keep in mind when making a slurry as it will have a significant effect on the coatweight of your coating once the solvent has been dried off. The solid content of your mixture can be estimated prior to the preparation of the slurry, but, to get a more accurate value, a moisture analyser such as the Ohaus MB120 (g) can be used. This will heat the sample to remove the solvent, while recording its mass, allowing for it to give a solid content weight %, once the mass of the sample stops decreasing.

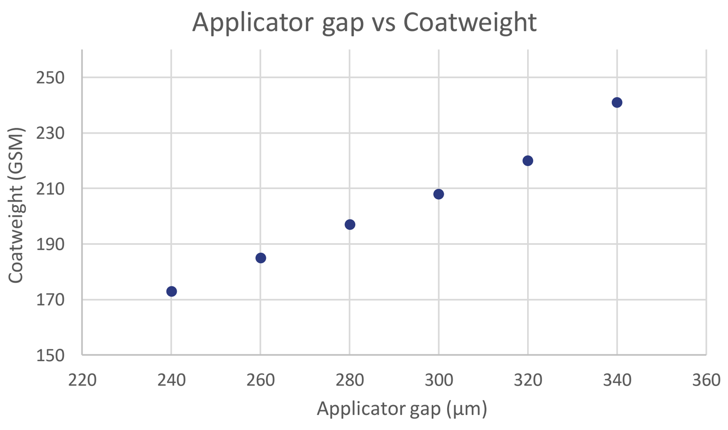
e

f

g

Coatweight

- The coatweight of a sample, which is recorded as GSM(g/m^2), will greatly affect the characteristics of a coating. A higher coatweight will give a higher capacity and energy density, although this also results in a lower power density.[3]



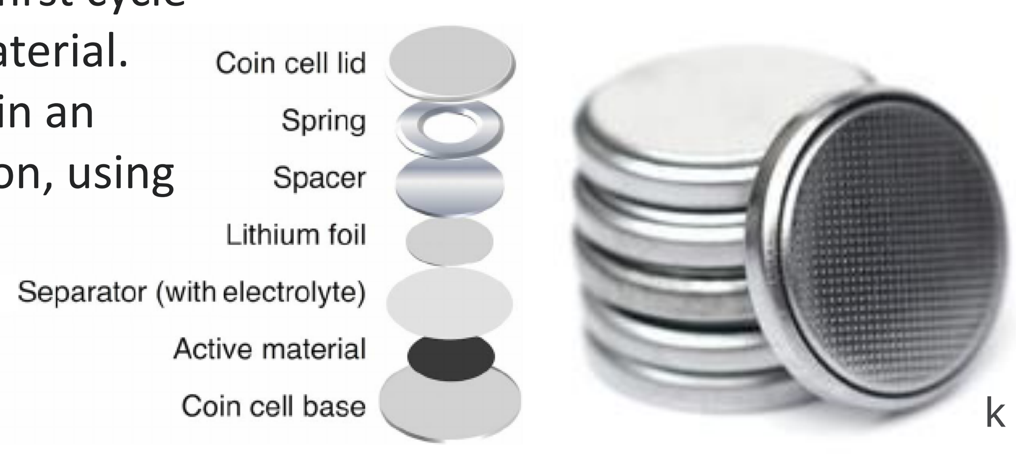
Coating Characterization

- A bend test (h) is a quick way to test the resistance of the coating to cracking or delamination from the current collector. Decreasing mandrel sizes are used to find the point where the coating will fail, which is important for flexibility in cylindrical cells.
- The zwickiLine (i) can be used to test the adhesion of the coating. It does this by slowly pulling the current collector away from the coating at a 90° angle while recording the force required. The higher the force that is required, the better the adhesion is. This can be used as an indication of how many cycles it will take for the coating to delaminate. Lower quantities of binder result in a lower force required to separate the coating from the current collector.
- Conductivity is a very important characteristic of a coating. This can be tested using a resistivity meter such as the Loresta-GX MCP-T700 (j). To test the conductivity of a coating, it is important that it is calendered to your target porosity, and coated on a non conductive material such as Mylar. Higher conductivity correlates to higher electrochemical performance.
- Half coin cells (k) can be made to test the discharge capacity and FCE (first cycle efficiency)[2] of a cathode material. The cell must be assembled in an inert atmosphere (eg. in argon, using a glove box), to keep the air-sensitive materials, such as lithium, from oxidizing.

h

i

j



k

Impact / Next steps

- New cathode materials with higher capacities than those currently available would facilitate the development of smaller or more powerful batteries, thereby potentially allowing different uses, such as in aeroplanes.
- Materials with lower cost may allow people who cannot afford current technology to be able to use the new innovations that they may need.
- Pollution could also be significantly reduced if new materials, which do not require such extensive mining, are found. This would help slow climate change.

References

[1] Kim, K.M. et al., 1999. Effect of mixing sequences on the electrode characteristics of lithium-ion rechargeable batteries. Journal of Power Sources, 83(1-2), pp.108–113.
[2] Goodenough, J.B. & Kim, Y., 2009. Challenges for rechargeable Li Batteries. Chemistry of Materials, 22(3), pp.587–603.
[3] Reynolds, C.D. et al., 2021. A review of metrology in Lithium-Ion Electrode Coating Processes. Materials & Design, 209, p.109971.

Intern bio

Ed Harrison is studying Chemistry at the University of Birmingham. Interested in energy materials science and how it can be used to improve everyday lives. Aspiring to have a career working in research and development of new battery materials.

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