Improved Mixing Performance Drives Innovation in Battery Development

Effective slurry mixing for electrode material has a significant effect on the performance of a lithium-ion battery fabricated downstream. A collaboration improved the slurry-mixing ability and improved the quality of the electrode materials

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The global shift towards cleaner and more sustainable energy sources has accelerated due to increased public demand, legislative measures, policy initiatives, and funding from both public and private sectors. This transition necessitates advancements in technology to reduce emissions and improve energy storage systems, particularly those that integrate renewable energy sources into the existing grid.

A key aspect of improving energy storage is high-performing lithium-ion batteries (LiBs), and a key player in the pursuit of battery technology innovation is the Battery Innovation Center Inc. (BIC; Newberry, Ind.; wwww.bicindiana.com), a non-profit public-private partnership. With its state-of-the-art facilities (Figure 1) and integrated approach to battery research, development, and testing, the BIC is at the forefront of energy storage technology and manages all aspects of the battery and energy storage lifecycle while creating a secure environment for the interchange of information and expertise. This article describes a collaborative project between BIC and original equipment manufacturer Charles Ross & Son Company (Hauppauge, N.Y.; www.mixers.com), where Ross mixers were used to improve the manufacture of electrodes for Li-ion batteries.

Electrode slurry: mixing matters

Battery cell fabrication consists of several steps, including electrode slurry mixing, electrode coating and drying, electrode calendering/densification, electrode slitting, cell fabrication, electrolyte filling, and electrochemical testing. Electrode slurry mixing is highly crucial in the early stage of the process because the quality of mixing directly impacts the overall performance and consistency of the final battery product. The proper ratio of active materials, binders, conductive additives and solvents must be thoroughly combined. Any solid agglomerates should be finely dispersed, while also taking care not to over-shear the slurry, which can negatively impact viscosity, stability and overall battery performance. Through optimal formulation and mixing, slurry rheology is tightly controlled to ensure that both cathode and anode can be applied effectively during the coating and electrode fabrication stages.

As the BIC facility expanded and its customer base grew, the team recognized the necessity to upgrade their smaller R&D laboratory-scale mixers. They sought the expertise of Charles Ross & Son Company, known for its industrial mixing, blending, drying and dispersion equipment. Founded in 1842, Ross Mixers is a leading supplier of mixers to the process industries, including the electronic and energy storage sectors.

Scale-up challenges

Prior to using Ross Mixers, the BIC laboratory depended on a benchtop overhead stirrer, capable of mixing batches under 100 mL, and a centrifugal planetary mixer with a 300-mL maximum working capacity. While these mixers are still being used for smaller experiments, they pose some challenges when scaling up to larger batches. The overhead stirrer was limited by an open-container design, which can lead to some material loss.

Cara Fagerholm, the senior engineer on the Advanced Battery Development team at the BIC, explains, “Mixing action inherently accelerates evaporation due to the increase in surface area for vapor exchange and rise in temperature. This evaporation may seem small, however, even minor adjustments in slurry solids content can cause significant changes in dispersion and viscosity, and therefore affect the coating quality of a material. Coating quality, in turn, impacts electrochemical performance, so it is important to have fine control of solids content in a closed container. Meanwhile, it is also ideal to reduce water uptake to avoid oxidation in LiBs, as this oxidation adds resistance and attenuates electrochemical performance. Therefore, a closed mixing vessel has a large impact on electrode quality.”

FIGURE 1. The Battery Innovation Center, located in Indiana, conducts research, development and testing of energy-storage systems

Though it provides a closed container, the centrifugal planetary mixer faces limitations in volume and relies on particle-particle interactions to generate shear for slurry dispersion. While effective for certain components, it falls short in dispersing some of the new formulations still undergoing development at BIC. This can impact the measurement of the active materials’ true capabilities.

Planetary disperser mixers

The BIC testing facility is now equipped with a pair of Ross PowerMix Planetary Disperser Mixers (PDMs). One unit has a volume capacity of 0.5 gal (Figure 2) and the other unit has a capacity of 2 gal.

The Ross PowerMix is a hybrid

planetary disperser designed to impart a combination of high- and low-speed agitation for mixtures that start out very fluid and eventually thicken up throughout the course of the batching process, making it well-suited for batching electrode slurries. Consisting of a planetary stirrer blade and a high-speed disperser blade, the planetary agitator revolves around a central axis while rotating on its own axis. The planetary blade sweeps material away from the vessel's sidewalls and bottom, carrying it to the saw-tooth blade. This speeds up solids wet-out and dispersion while maintaining material and temperature consistency throughout the batch at all times.

The robust mixing capabilities and advanced design of the Ross PowerMix allow BIC scientists and engineers to consistently produce homogeneous slurries with excellent particle dispersion, regardless of battery chemistry.

Mixing trials performed at BIC are essential for fine-tuning electrode slurry formulations. Initial steps in optimization often focus on the mix sequence and powder ratios to ensure good dispersion and high active material (AM) content. A proper mix sequence is necessary to ensure the successful incorporation of powders and assists in monitoring for quality assurance and quality control (QA and QC) metrics along the way. High amounts of AM are preferable, as the AM is what contributes capacity to a cell, and a higher amount of AM allows the cell to demonstrate a higher capacity per total cell mass. The typical next step is to further optimize the formulation by adjusting solids content. The removal of solvent during the coating process for electrode drying is highly energy-intensive, and it can cause damaging stresses on the surfaces of the electrode and at the current collector interface. Hence, a low solvent content or, consequently, a high solids content is a desired outcome.

Since adding the Ross PowerMix, the BIC can efficiently produce slurry for roll-to-roll coating in a single batch, ensuring homogeneity throughout the mixing process. This single-batch approach is preferred over combining multiple batches from the overhead stirrer and centrifugal mixer as it minimizes variations and enables the production of consistent and higher-quality electrodes and cells.

BIC

FIGURE 2. The Ross PowerMix 0.5-gal capacity mixer produces homogeneous slurries for battery electrode development

"Further, the use of the Ross mixer aids to improve quality control with repeatability and reliability. This is critical for R&D processes where there can be divergent rheological QC metrics for the same degree of dispersion, depending on input materials," Fagerholm explains, "Typically, we can drastically increase the amount of energy input in our Ross mixers, as permitted by the cooling jacket system and capability for overnight mix profiles. This increased mixing energy can improve the dispersion and increase slurry viscosity as particles separate and all possible particle surfaces become solvated, reducing 'free NMP' [*N*-methylpyrolidone]. However, depending on the surface energies of the materials involved, better-dispersed slurries can have a higher or lower rheology result. Surprisingly, cathode active materials can attract other solid particles in the slurry and create more 'free NMP' in the system to reduce the viscosity."

Meanwhile, BIC also observes that polymer strands can become better aligned in the Ross mixer and drop viscosity for shear-thinning binder systems. The use of a single batch of slurry eliminates the potential inconsistencies that can arise from batch-to-batch variations. This improved uniformity facilitates the precise control of key parameters, such as composition and viscosity. The higher-power, semi-automated mixing apparatus also enables BIC to more definitively obtain and monitor these QC parameters, which are critical for the performance and reliability of the final battery cells.

Another benefit is the ability to translate results and rheology upon scaleup. With well-designed materials and mixing order-of-operations, the BIC is often able to seamlessly replicate outcomes from smaller to larger mixers. This has accelerated their R&D timelines, allowing swift scaleup from multiple small-scale slurry trials to the Ross systems with minimal adjustments.

The Ross PowerMix's vacuum-capable design is yet another advantage. Establishing a vacuum in the mixing vessel efficiently removes any air introduced during mixing. Air trapped in the slurry can cause bubbles to form during the coating process, especially on the surface of the current collector. These defects decrease the quality of coated electrodes and can expose bare metal foils during the battery charge and discharge process, which result in internal shorts within the battery. This also causes unwanted side reactions that reduce the capacity and lifecycle of the cell and may result in catastrophic damage during electrochemical testing.

With the agitators vertically oriented, the Ross PowerMix has no shaft seals, bearings, packing or stuffing boxes submerged in the liquid zone. Agitators are raised and lowered into and out of the mix vessel by a hydraulic lift, enabling easy access for cleaning. The Ross mixers at BIC are operated from a 10-in. touchscreen, and the PLC-based controls can be programmed to run mixing, heating and vacuum sequences, which maximizes overall productivity by improving batch-to-batch consistency and reducing operator errors while accelerating product changeovers. ■

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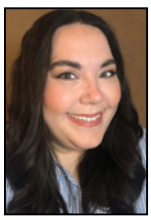

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