

The stability of coating slurries used for the production of anodes and cathodes was tested using the dispersion stability analysis system MultiScan MS 20. Analysing the time-dependent and position-dependent transmission and backscattering intensity of different slurries, unstable slurry formulations could be identified within a short period of time which is valuable information in the development and optimisation of battery slurries.



Fig. 1 left: Battery pack for electric vehicle
 right: Battery slurry 1 in sample container after 26 hours

Background

Electro mobility is one of the key elements for climate-friendly transportation and has the potential to render our environment cleaner as well as to improve quality of life especially in urban areas. The success of electro mobility strongly depends on the affordability and efficiency of the utilised battery systems. Tremendous efforts are undertaken in labs all around the world aiming at the improvement of different components of the batteries, such as the anodes and cathodes [1].

Lithium batteries are the most widespread used mobile battery systems [2]. Their electrodes are made up of multi-component mixtures that are manufactured from dispersions of micro- or nano-scaled powders in highly viscous polymer solutions. These 'coating slurries' contain a large percentage of solid particles of different composition, size and shape.

In order to facilitate the electrode production and guarantee products of reproducible quality it is crucial that the coating slurries are homogeneous and stable regarding the distribution of their ingredients.

Hence, during the development process of the coating slurries, it is essential to study the dispersion stability. Challengingly, the separation of individual components is very often invisible to the naked eye for weeks, or even months, which makes technical help a must have for a fast and timely product development process.

The dispersion stability analysis system MultiScan MS 20 from DataPhysics Instruments (Fig. 2), with its matching software MSC, is the ideal partner for the thorough stability optimisations required in the development of nano-particle based battery slurries. It is able to detect even slightest changes within dispersions and thus allows looking into and evaluating any occurring separation processes fast and objectively. Moreover, a dispersion stability analysis with the MS 20 can also provide information on possible destabilisation mechanisms which is helpful to eventually eliminate the instabilities.

A study of two different nano-particle based battery coating slurries with the MS 20 and a comparison of the results will be presented throughout this application note.

Experiment

For analysing a dispersion with the MultiScan MS 20, the dispersion is filled into a standard sample container (max. volume 27 ml) which is then placed into one of the system's "Scan Towers". The towers incorporate a scanning unit, composed of a transmission and a backscattering LED along with a detector, which moves up and down the vertical side of the sample container (along the z-axis) in custom-defined time intervals. This allows to detect the transmission and backscattering intensities both position-resolved and time-resolved, which is represented as multiple intensity profiles in intensity-position diagrams in the MSC software (see Fig. 3, Fig. 4 and Fig. 5).

In the experiment described here the stability of two battery slurries with different formulations was studied. For this purpose 20 ml of each battery slurry was poured into a standard sample container and put into a scan tower in which temperature had been set to $T = 25\text{ }^{\circ}\text{C}$. A measurement routine was set up which scheduled scans every 5 min for a total measuring time of 26 hours (slurry 1) or 120 hours (slurry 2), respectively. The scanned z-axis range was between 0 mm (bottom of the sample container) and 50 mm (fill level).

Fig. 1 right shows the sample container filled with unstable battery coating slurry 1 at the end of the experiment.



Fig. 2: Dispersion stability analysis system MultiScan MS 20 from DataPhysics Instruments

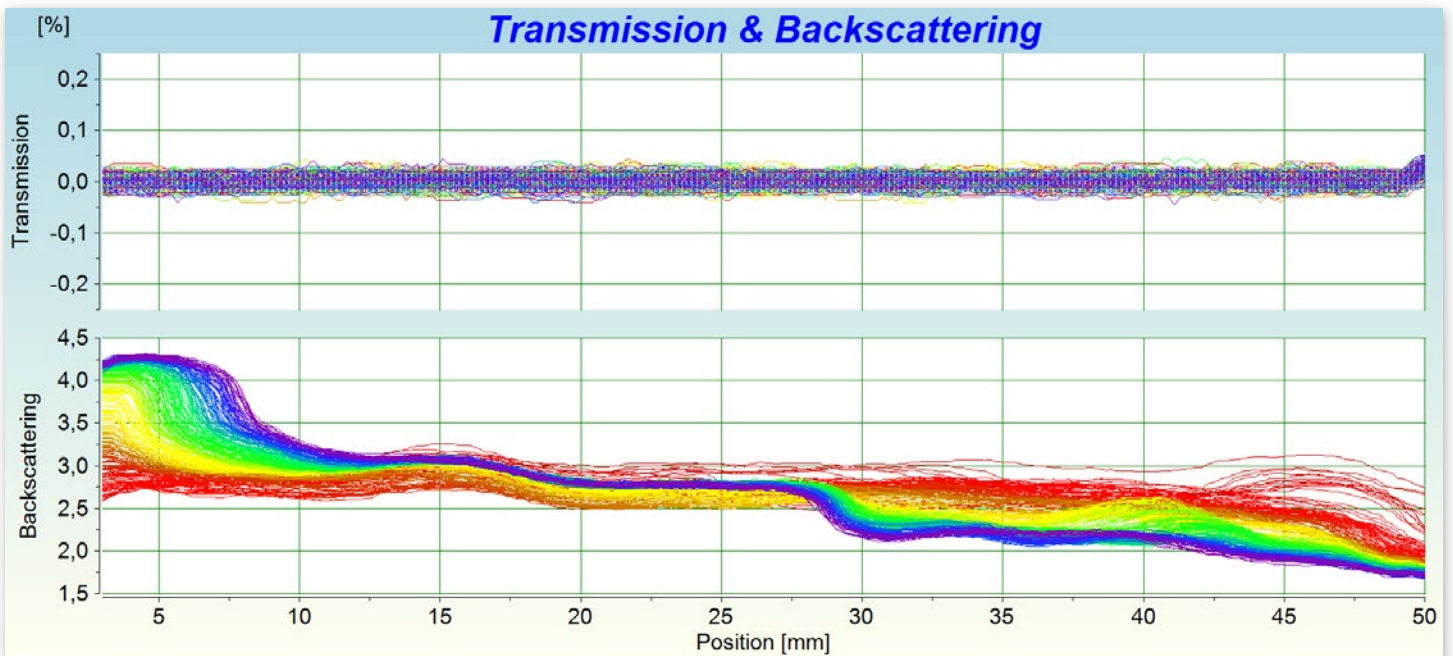


Fig. 3: Transmission (top) and backscattering (bottom) intensity vs. position diagrams for battery slurry 1, with intensity profiles colour-coded from red to violet from the first to the last scan, respectively

Results

Fig. 3 shows the plots of the transmission and backscattering intensities against the position for battery slurry 1. The colour-coding indicates the time at which the individual intensity profiles were recorded (red: $t = 0$ s, to purple: $t = 26$ h).

The transmission profiles of slurry 1 (Fig. 3, top) show a constant mean intensity value of $I_{tr} = 0$ %, which does not change throughout the whole experiment. This can be explained by the turbidity of the battery coating slurry that prevents the transmission of any incident light.

The backscattering diagram (Fig. 3, bottom), on the other hand, shows a clear time-dependent and position-dependent change of the intensity signal.

This indicates that battery slurry 1 is not stable over the investigated time period but some destabilisation processes are occurring. This becomes even more obvious in a relative plot where the change of the backscattering intensity compared to the one of the very first scan is shown (see Fig. 4).

Looking closer at the shape of the backscattering profiles in Fig. 4 one can see an increase of the backscattering at the bottom of the sample container along with a backscattering decrease at the top of the sample. This indicates a sedimentation process with a sedimentation layer building up at the bottom and the sample clearing up from the top (see Fig. 1 right).

Hence, a possible further analysis step could be the determination of the sedimentation velocity using the Migration Front analysis option of the MSC software.

For the second studied sample, battery slurry 2, the situation looks completely different. As can be seen in Fig. 5, for battery slurry 2 both the transmission and the backscattering intensities did not change during the whole measurement time of 120 hours. This indicates that slurry 2 is very stable and hence from this perspective a great candidate for battery coating.

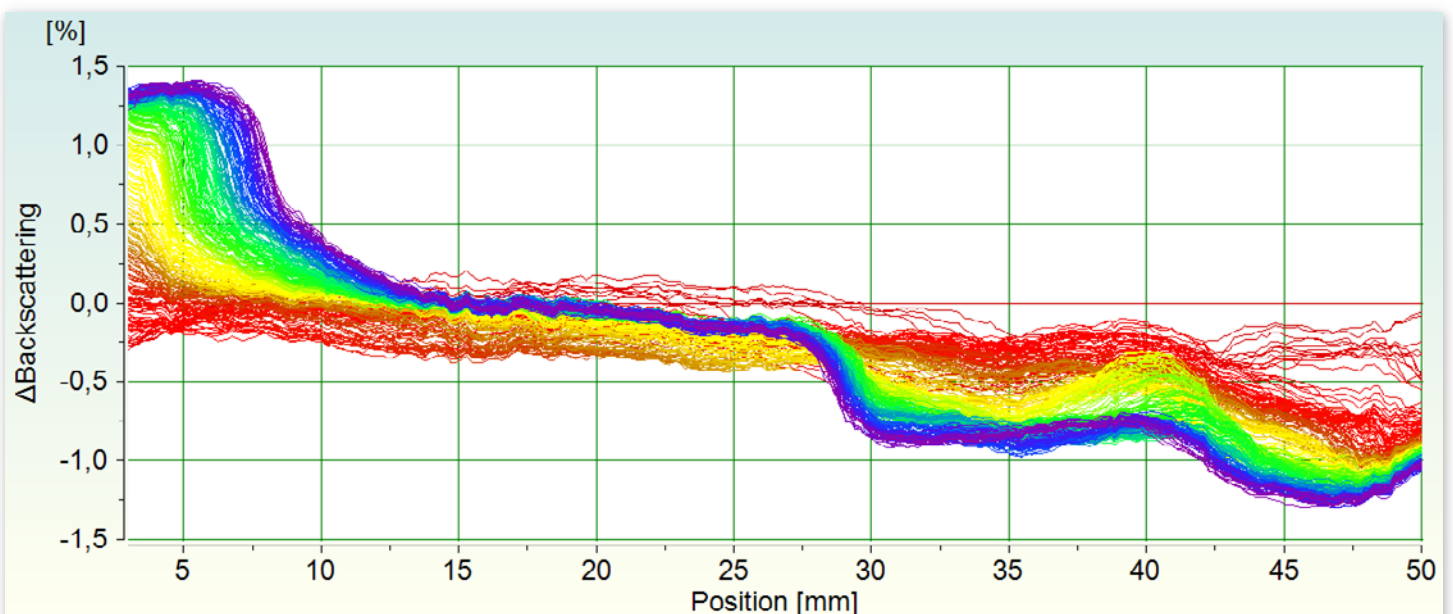


Fig. 4: Relative backscattering intensity vs. position diagram with profiles illustrating the intensity relative to the intensity measured at $t = 0$ s

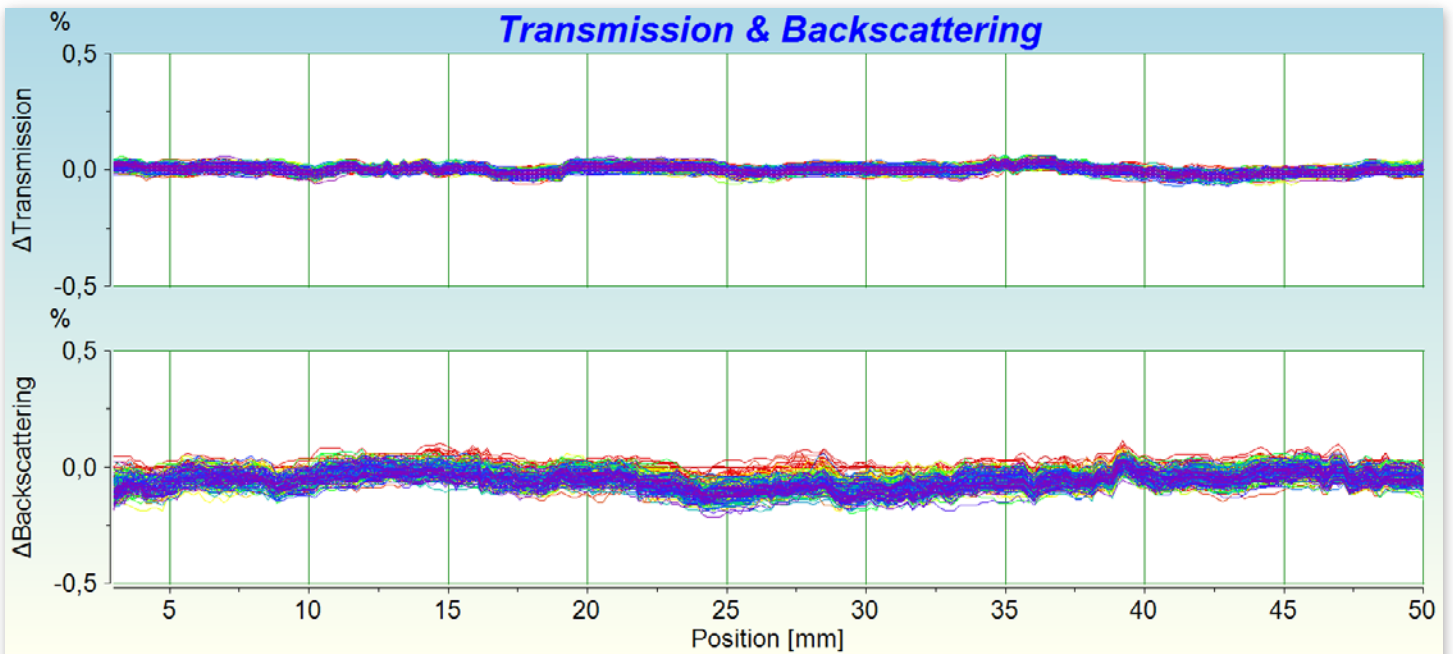


Fig. 5: Relative transmission (top) and backscattering (bottom) intensity vs. position diagrams for battery slurry 2 (intensities relative to the one measured at $t = 0$ s)

Summary

Using the dispersion stability analysis system MultiScan MS 20 from DataPhysics Instruments and the corresponding MSC software the stability of two different battery slurries was studied and compared. Recording transmission and backscattering intensity profiles for a period of 26 and 120 hours, respectively, one of the slurries (sample 2) could be identified as stable while the other one (sample 1) clearly turned out to be unstable.

Due to the turbidity of the two slurries this was not seen in the transmission data, but became directly obvious looking at the (relative) backscattering profiles which show distinct changes for battery slurry 1, after a couple of hours, while for slurry 2 there are no changes during the whole measuring time of 5 days.

Due to the shape of the evolving backscattering profiles of battery slurry 1 one can furthermore deduce that the predominant destabilisation process is apparently sedimentation as a migrating sedimentation front and a growing sedimentation layer are seen.

The opportunity to observe position-resolved and time-resolved even smallest changes of a sample's backscattering and transmission within a very short period of time enables the producers of battery coating slurries (as well as producers of all kinds of dispersions) to fast and objectively carry out stability analysis. Thus, the dispersion stability analysis system MultiScan MS 20 from DataPhysics Instruments helps to quickly anticipate long term stability and thus guarantees a time and cost efficient product development.

References

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- [2] Brodd R.J. (2009) Synopsis of the Lithium-Ion Battery Markets. In: Yoshio M., Brodd R. J., Kozawa A. (Eds.) *Lithium-Ion Batteries*. Springer, New York, NY