CURINSCAN Expert

In-situ drying monitoring of battery slurries

INTRODUCTION

One of the world's biggest challenges is to maintain sustainable growth while preserving natural resources. Forecasts predict that we will be using 30% more energy in 2040 than we do today. These changes are likely to impact the battery market due to the booming automotive industry, rising demand for energy storage systems, widening applications in the motive industrial sector, and growing consumption of rechargeable batteries in consumer electronics...[1]. The expanding market requires new more efficient battery types (for example: higher energy density and larger capacities).

Battery slurries | **Drying times** | **Temperature & thickness influence**

HOW IT WORKS

CurinScan is based on **Nanoscale Mobility Analysis (NMA)** and measures the particles' (polymers, aggregates, pigments...) Brownian motion, thanks to an optical method.

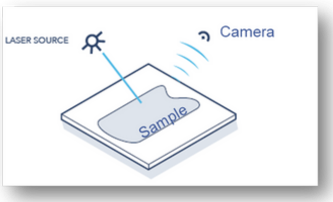

Figure 1: Measurement concept

During the film formation or curing process, the nanoscale mobility of particles changes due to the material properties' evolution, for example from liquid to solid.

When a material (coating, film, adhesive...) presents an important microscopic dynamics activity (liquid-like), the structures present an important Brownian motion directly correlated to the visco-elastic properties of the material.
Thanks to a dedicated image analysis algorithm, it is possible to determine a characteristic frequency, the **microscopic dynamics (µD or mD),** which directly correlates to the speckle image fluctuation on the camera.

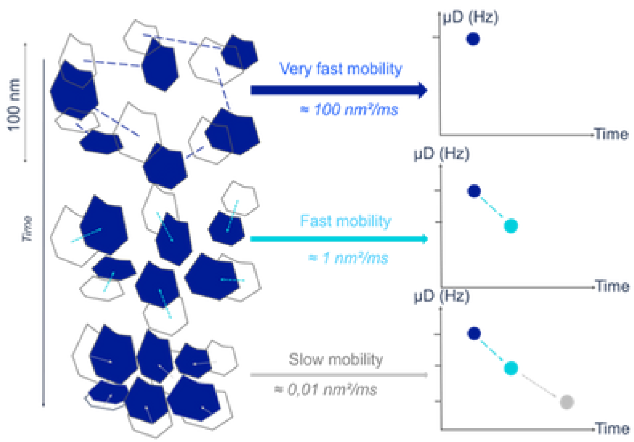

Figure 2 : Schematic representation of the mobility (µD) evolution during typical drying/curing process versus time

When the sample is liquid (early times), it presents a high level of mobility (µD). This mobility will be decreasing over time during the drying/curing process.

PROTOCOL & METHOD

The experiments were done on NANOMYTE BE-45 (NCA) slurry, Lithium Nickel Cobalt Aluminum Oxide powder from NEI Corporation [3] over 8 hours and at 2 different temperatures and by applying layers of different thicknesses. The aim is to define the optimal conditions for manufacturing.

RESULTS

Detection of characteristic "Set-to-touch Time": Influence of the thickness and temperature on the drying process

Figure 3 shows the microscopic dynamics (mD) versus time for different sample thicknesses (100 and 250µm) and different temperatures (50 and 100°C). From the dramatic decrease in mobility (mD), a characteristic time can be determined. Defined as set-to-touch time, it is the time needed for the evaporation of the solvent, when the film stops flowing and will not adhere to a light touch.

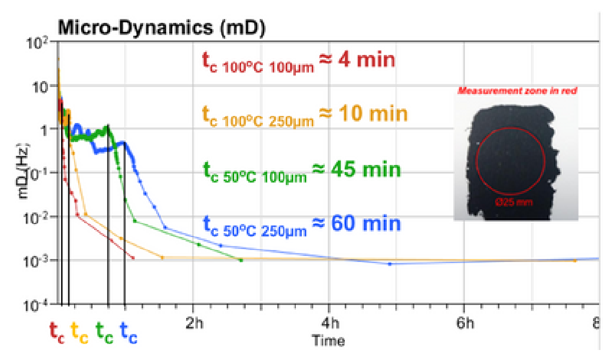

Figure 3: Micro-dynamics function of time for cathode slurry at different temperature and different coating thickness

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The characteristic time TC is influenced by the thickness and temperature. At temperature (50°C for example), the TC of the thinner sample (100µm, green) is shorter than the TC of the 250 µm thick sample (blue) by 15 minutes. Additionally, increasing the temperature from 50°C to 100°C shortens the characteristic time by a factor of 6 to 10 depending on the applied thickness

The instrument provides unique data on the battery's slurry kinetics that enables quantifying the relation between key parameters in the drying process. Knowing the dependencies between these 3 parameters (time, thickness, and temperature) guides to tuning the formulation or other parameters to optimize the fabrication process.

Detection of characteristic "Drying Time": Influence of the clear thickness and temperature on the drying process

Another important piece of information is the **exact determination of the final drying time**, the time required for the loss of volatile components so that the material will no longer be adversely affected by a large twisting force. To do so, we will focus on the time when the mD reached a plateau at 10-3 (Hz) (fig. 4). When the mD level reaches a plateau at 10-3 (Hz) the sample dynamics contribution is almost negligible, indicating that the sample is completely dry.

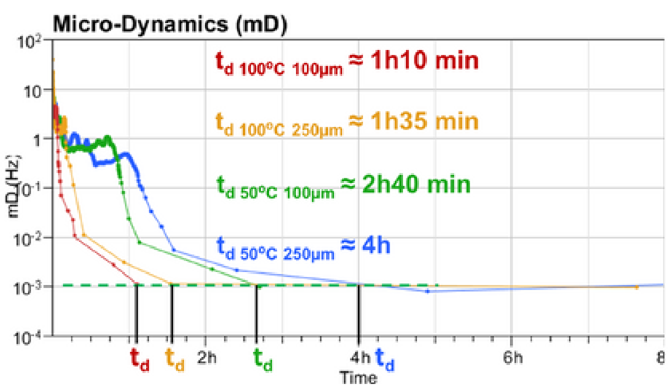

Figure 4: Curing time as a function of the curing temperature for an epoxy adhesive

Figure 4 shows how the drying time is influenced by the thickness and temperature. At the same temperature (100°C for example), the drying time Td of the thicker sample (250µm, yellow) is longer than the Td of the sample 100µm thick (red) by 25min. At 50°C, this difference increases to 1h20 min. Despite the analysis time of 8 hours, the results can be analyzed after 4 hours.

Drying Time Ranking

To facilitate the ranking, the software allows to computation of the microscopic dynamics evolution, mDE (fig. 5). The curves (fig. 5) correspond to the areas under each mD curve (fig. 4) on a selected time window. The mDE is directly correlated to the drying speed. The faster the mDE reaches 100%, the faster the drying.

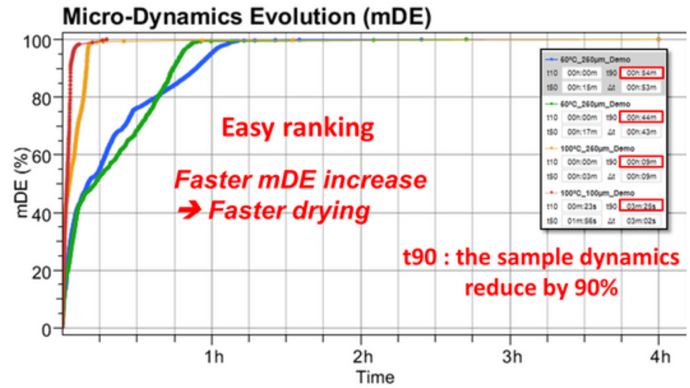

Figure 5: Micro-dynamics evolution function of time for cathode slurry at different temperature and different coating thickness

Figure 5 shows a ranking in agreement with the drying times determined for the different samples (fig. 4). The software allows also to determine another characteristic time **'t90'** where the slurry's microscopic mobility is reduced by **90%** (inset figure 5). For this slurry, the **'t90'** is very close to the time needed for the evaporation of solvent identified in figure 3 **('t90' ≈ 'Td')** due to the very rapid decrease in mobility.

Studied slurries can have very different drying kinetics depending on applied conditions (thickness or temperature). The variation can be significant and considerably impacts battery performance if these parameters are not optimized.

CONCLUSION

We have seen here how the CurinScan can help identify characteristic drying times for such slurries, as a function of key parameters or formulation variants. This presents a new ***in-situ*, non-invasive and handy** method for a better understanding of different materials, allowing:

- The **monitoring** and **precise knowledge** of the **curing** and **drying kinetics** (from minutes to hours)
- The **determination of the characteristic times** of the film-forming process
- The evaluation of the impact of **formulation** on the film formation
- The **optimization** of the manufacturing protocol according to **different substrate materials**

References :

[1] https://www.inkwoodresearch.com/reports/global-battery-market/
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[4] PINE, WEITZ, et al. Diffusing-wave spectroscopy: dynamic light scattering in the multiple scattering limit. 1990.