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Ex-Situ Surface Analysis of Electrode-Material with Diffuse Reflectance Spectroscopy

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Motivation

Electrode-wide inhomogeneity remains unexplored

- Chemical and morphological surface analysis help revealing causes for battery degradation and failure
- Common optic-emission based analysis methods (EDX, XPS, GC) are limited in sample size, throughput and come at high equipment costs
- An surface-analysis of the whole sample (i.e. electrode-sheet) is nearly impossible

→ *Limitations of common surface analysis methods do not allow a revelation of inhomogeneity over full electrode sheet*

Where the community already has an eye on

- Grade of lithiation has massive impact on the anodes colour [1][2]
- Li-plating can be observed as silvery-greyish precipitation on anode surface [3][4]
- Degradation of the anode has a spatial inhomogeneous appearance [5][6]

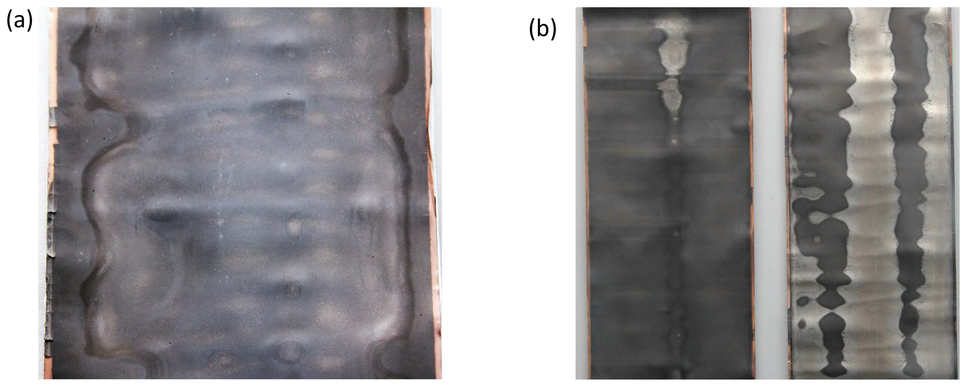

Figure 1: Photograph of aged LG Chem. INR18650-MJ1 anode-sheets after cell-opening under argon-atmosphere. Not only li-plating can clearly be observed (b), but also further inhomogeneous precipitation (a).

→ *Ageing effects and its inhomogeneous appearance over electrode-sheet can already be observed, but are not captured yet*

Principle of Methodology

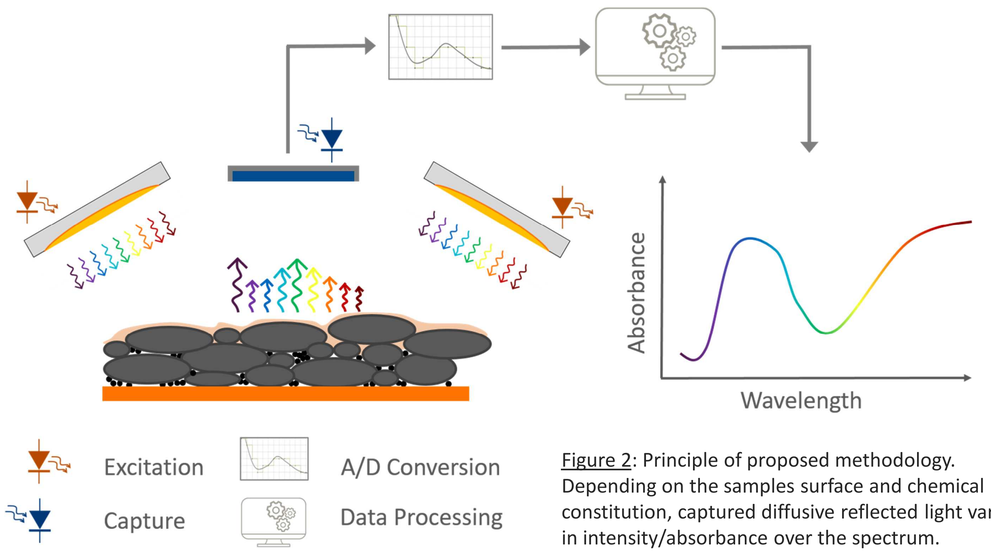

Figure 2: Principle of proposed methodology. Depending on the samples surface and chemical constitution, captured diffusive reflected light vary in intensity/absorbance over the spectrum.

How the surface is analysed

- Broadband light-source is used to illuminate sample
- Depending on morphology and chemical constitution, light is either diffusely reflected or absorbed. Transmission is not taking place due to the solid nature of the sample.
- The intensities of different spectra is captured by a photo-sensor and analysed in comparison to a reference measurement under same conditions.
- Thereby visual differences over aging can easily be captured and quantified

Experimental

- Illumination is realised via warm-white LEDs (3000 K)
- Sensor is capable of capturing intensities for 18 different wavelengths
- 4 anodes (1 pristine + 3 from aged single-layer pouch cells) examined
- Surface area (5 cm x 5 cm) is separated in 25 equal sections (1 cm x 1cm), whereas only the inner 9 sections are considered for comparison (Fig. 3(f))

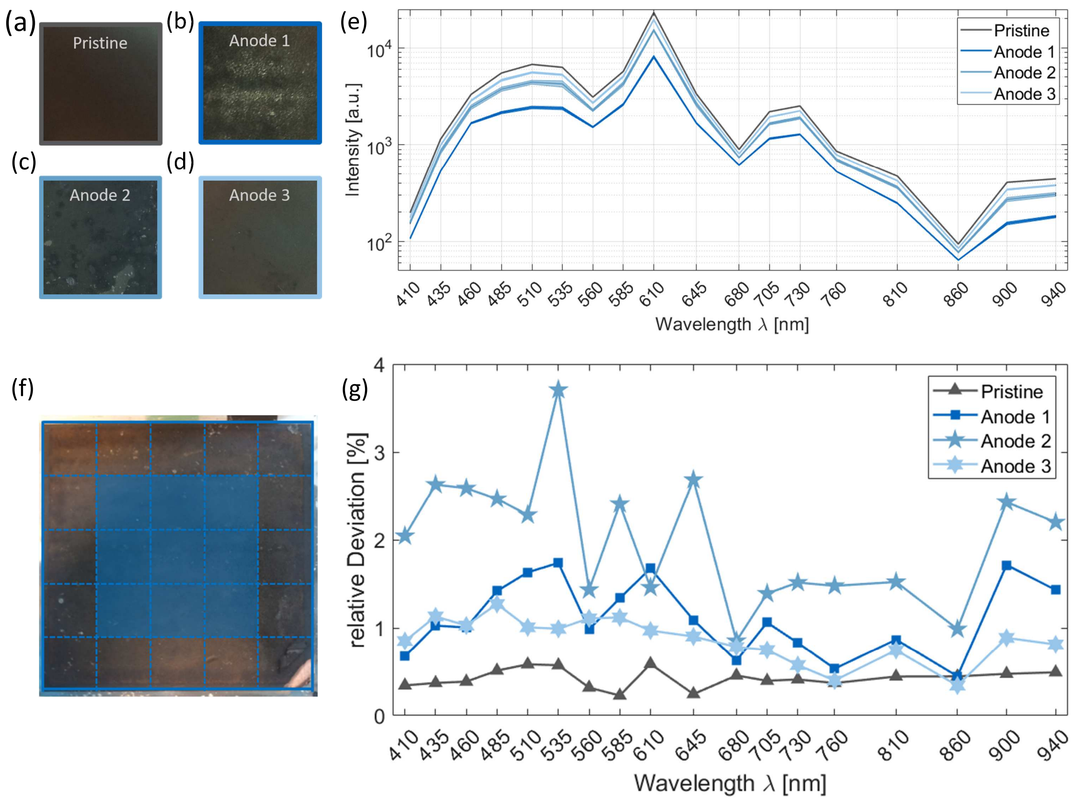

Figure 3: Results of the diffusive reflectance spectroscopic examination of for SLP anodes. 4 anodes have been investigated where (a) shows pristine material and (b) – (d) show anodes with different ageing behaviour. For investigation spectra of the 9 inner sections have been considered (f). The anodes can clearly be distinguished in the mean of their spectra (e). The pristine shows the lowest relative standard deviation, whereas spectra of Anode 2 shows the highest relative standard deviation (g).

Results

- Different anodes can clearly be distinguished in their spectra (Fig. 3(e))
- Pristine material shows highest reflectivity and lowest relative Deviation within examined sections (Fig. 3(g))
- Anode 2 show highest deviation in intensity, which correlates with highest inhomogeneous visual appearance (see Fig. 3(c))
- For wavelengths between 435 nm to 645 nm relative differences in reflected intensities to the mean of single sections (Fig. 4(a)) differ up to 17% (Fig. 4(b))

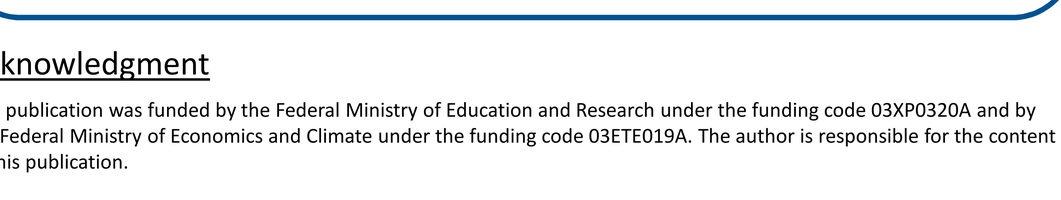

Figure 4: Inhomogeneous precipitation on surface of Anode 2 (a) is also visible as high deviation in measured reflective spectra (b).

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