CHADA

Characterisation Data and description of a characterisation experiment

For Scanning Microwave Microscopy (SMM) in liquid

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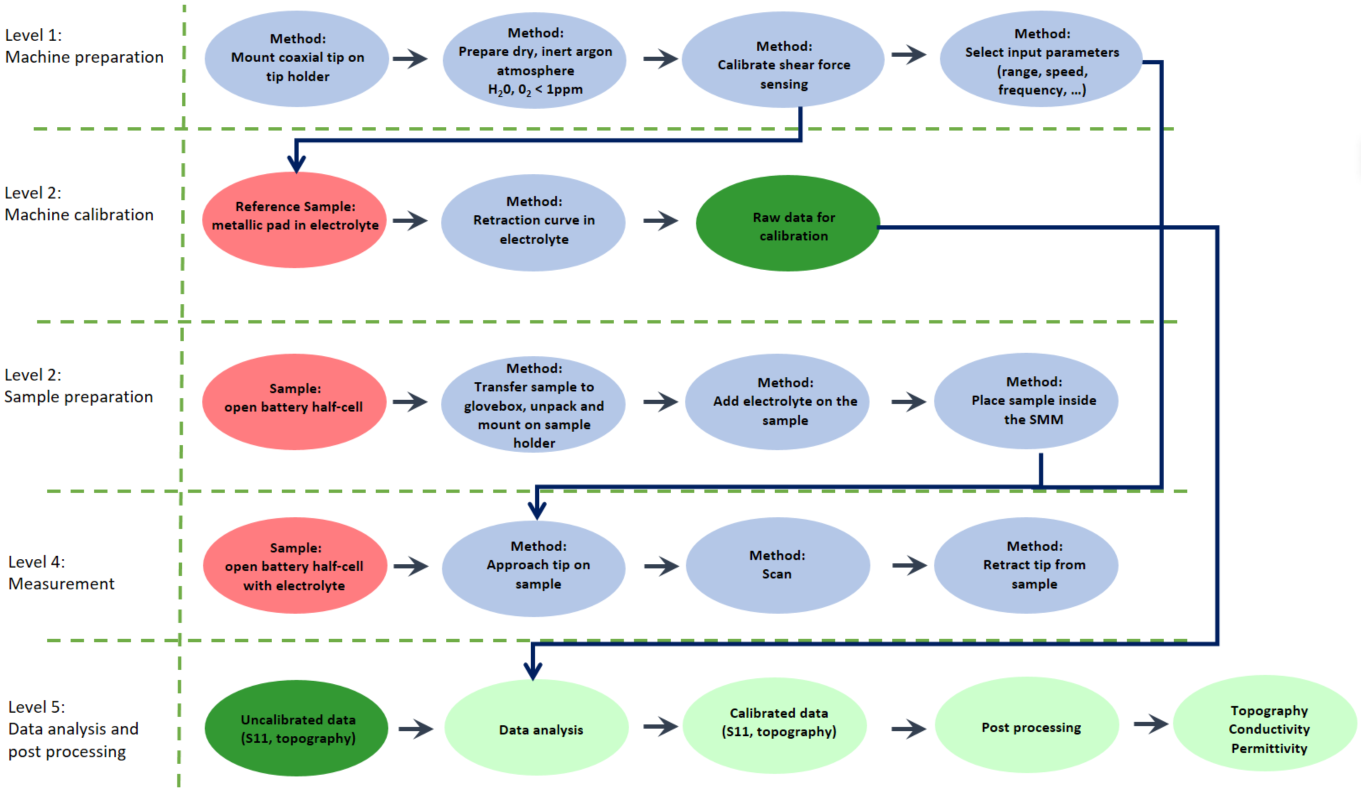
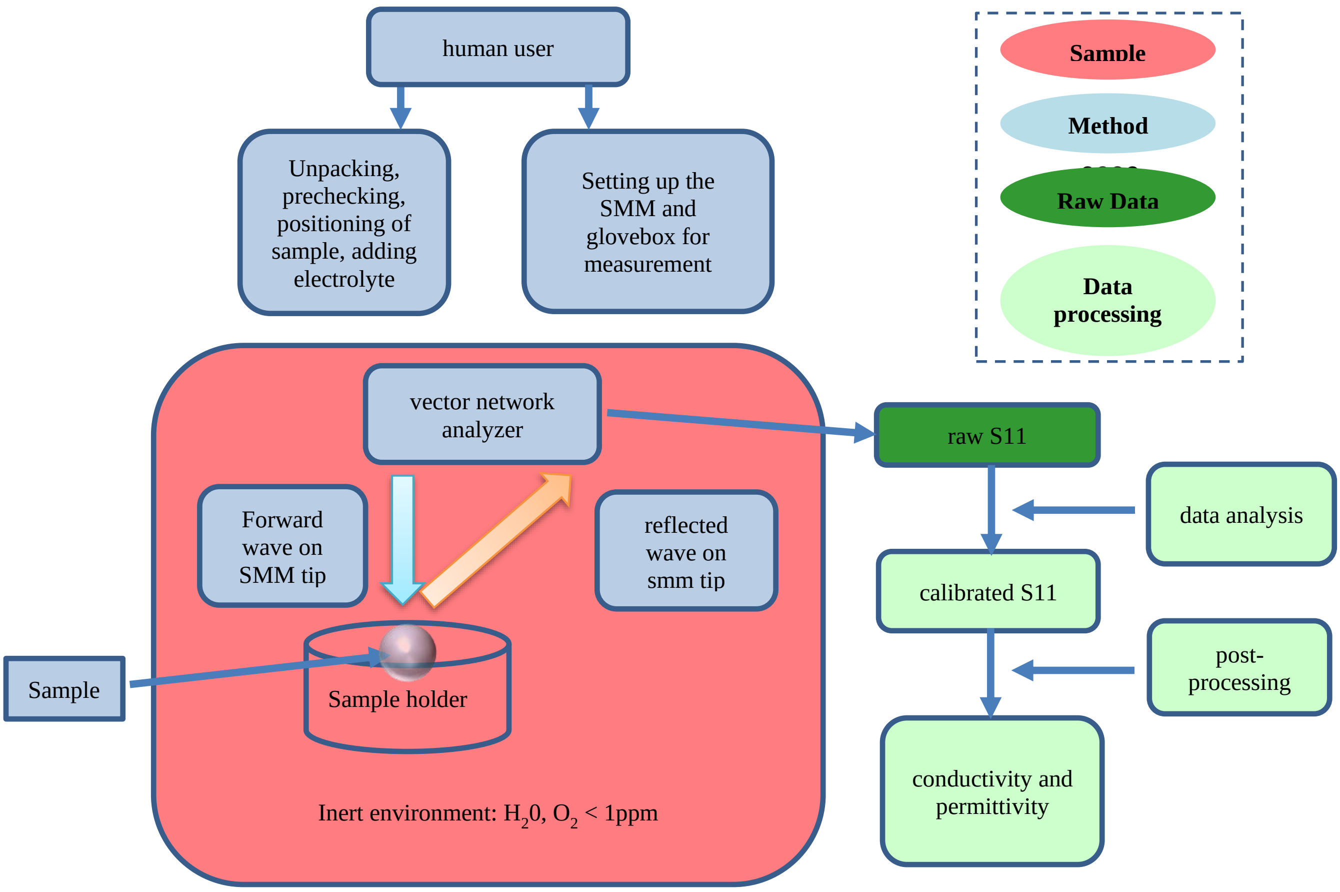
Used in NanoBat (H2020)

Overview of the Characterisation

1	Sample	Open Li-Ion battery half-cells in inert, dry atmosphere (argon)
2	Chain of methods Method	SMM prep 1: Coaxial tip is mounted onto the tip holder
		Glovebox prep: Argon filled glovebox is prepared to guarantee dry, inert atmosphere
		SMM prep 2: Feedback-Loop for height control is calibrated
		SMM prep 3: Selection of all input parameters
		Sample prep 1: Half-cell is transferred to the glovebox, unpacked and glued onto the sample holder
		Sample prep 2: Electrolyte is applied onto the electrode
		Sample prep 3: Sample is placed inside the SMM
		Approach: Tip is brought in contact with the sample
		Measurement: 2D Scan according to the input parameters
		Retraction: Tip is retracted from the sample
3	Data publication	Open innovation environment (established during this project) and Zenodo (https://zenodo.org)
4	Access conditions	Open access for raw data. Can be read using Gwyddion (http://gwyddion.net, GPL)
5	Workflow of the characterisation	Raw data is acquired by following the chain of methods described in point 2. The measured S11 parameter is then calibrated using a three-term error model. Measured topography data does not require calibration. Finally, the calibrated S11 is post-processed to obtain conductivity and permittivity. The topography data is transferred to length units.

Workflow picture





1. Sample		
1.1	USER	Human users (some to various years of expertise in scanning microscopy), no automation of the test
1.2	User case (sample specifications)	Open Lithium-Ion battery half-cells. Sample dimensions approx. $30x30x1mm^3$. The electrode is glued onto a sample holder and brought to an argon filled glovebox. The electrolyte is applied onto the electrode within the glovebox.
1.3	Specimen	Battery heterogeneous material (solid electrode and liquid electrolyte)
1.4	Testing environment	Inert gas environment, ambient pressure, H_2O and O_2 < 1ppm. Low noise and vibrations.
1.5	Material	Copper foil with SEI layer on top

2. Method		
2.1	Sample/probe physics of interaction	A high frequency (1-50GHz) radio signal is transported via tip, penetrates the sample and is back-reflected. The amplitude and phase of the reflected signal depends on the electrical properties of the sample.
2.2	Volume of interaction	A hemisphere with a radius of approx. 100nm (depending on the tip quality)
2.3	Equipment setup	Coarse X,Y,Z positioner, nano-positioner for scanning in X,Y. Nano-positioner in Z for tip-sample-distance control. Distance control loop measuring shear force with a piezo shaker and a piezo microphone. Vector Network Analyzer to generate and measure the radio wave.
2.4	Calibration	Shear force sensor: Lock-in technique on the loaded resonance frequency. S11 calibration: Retraction curve from a metal pad inside the electrolyte.
2.5	Probe	Coaxial tips produced by METAS; tip radius as good as 200 nm
2.6	Detector	The vector network analyzer (VNA) measures the amplitude and phase of the S11 parameter.
2.7	Signal	Complex S11 parameter Z Piezo sensor voltage.
2.8	Time lapse	Prep: Sample preparation and machine setup 1-2 hrs Measurement: 15min – 5hrs, depending on the resolution, scan size and probed frequency bandwidth.
2.9	Testing Input parameters	Probing radio frequency signal: power, frequency, signal bandwidth, number of RF pulses to average per pixel Scan: size and resolution Tip-Sample control: feedback-loop gains and setpoint.
2.10	Main acquired channels	Topography of the sample, amplitude and phase of the S11 parameter.

3.	**Raw Data**	
3.1	Raw Data	2D maps in binary (.dat) and Gwyddion native data file (.gwy) format of the topography and S11 (phase and amplitude)
3.2	Data acquisition rate	up to 140px/s

4.	**Data Processing**	
4.1	Main data filtering processes	The intermediate frequency bandwidth (IFBW) defines the pulse width of the probing signal. A high IFBW probes the sample over a wider frequency range and the measured response is therefore an average (convolution) over this range. Additionally, the raw data is an average over the number of pulses specified in the input parameters.
4.2	Main data analysis procedures	Calibrated S11 is obtained via a three-term error model.
4.3	Main processed channels	Topography, amplitude and phase of S11
4.4	Data processing through calibrations	*The calibrated topography is obtained by mapping the piezo sensor voltage to the corresponding piezo expansion.* The calibrated S11 parameter is used to calculate conductivity and permittivity using the results from the retraction curve described in 2.4
4.5	Properties (elaborated data)	