CHADA

Characterisation Data and description of a characterisation experiment

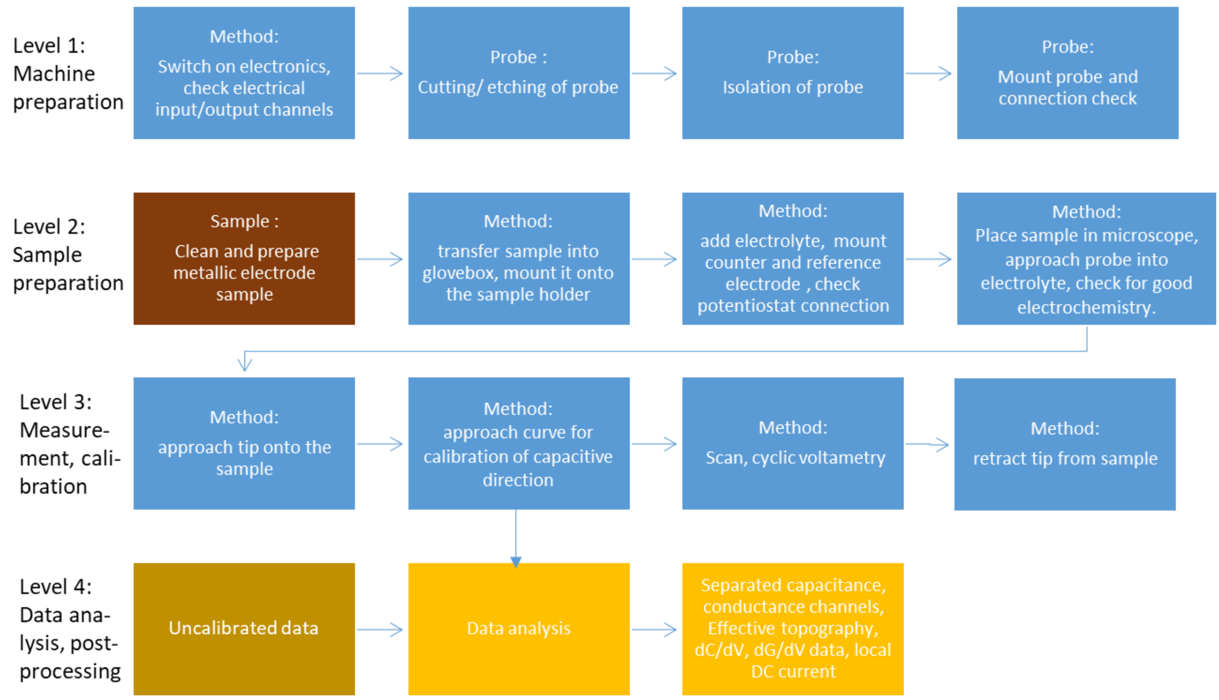
For broadband impedance microscopy (bb-IM) in electrolyte environment and a potentiostatic control

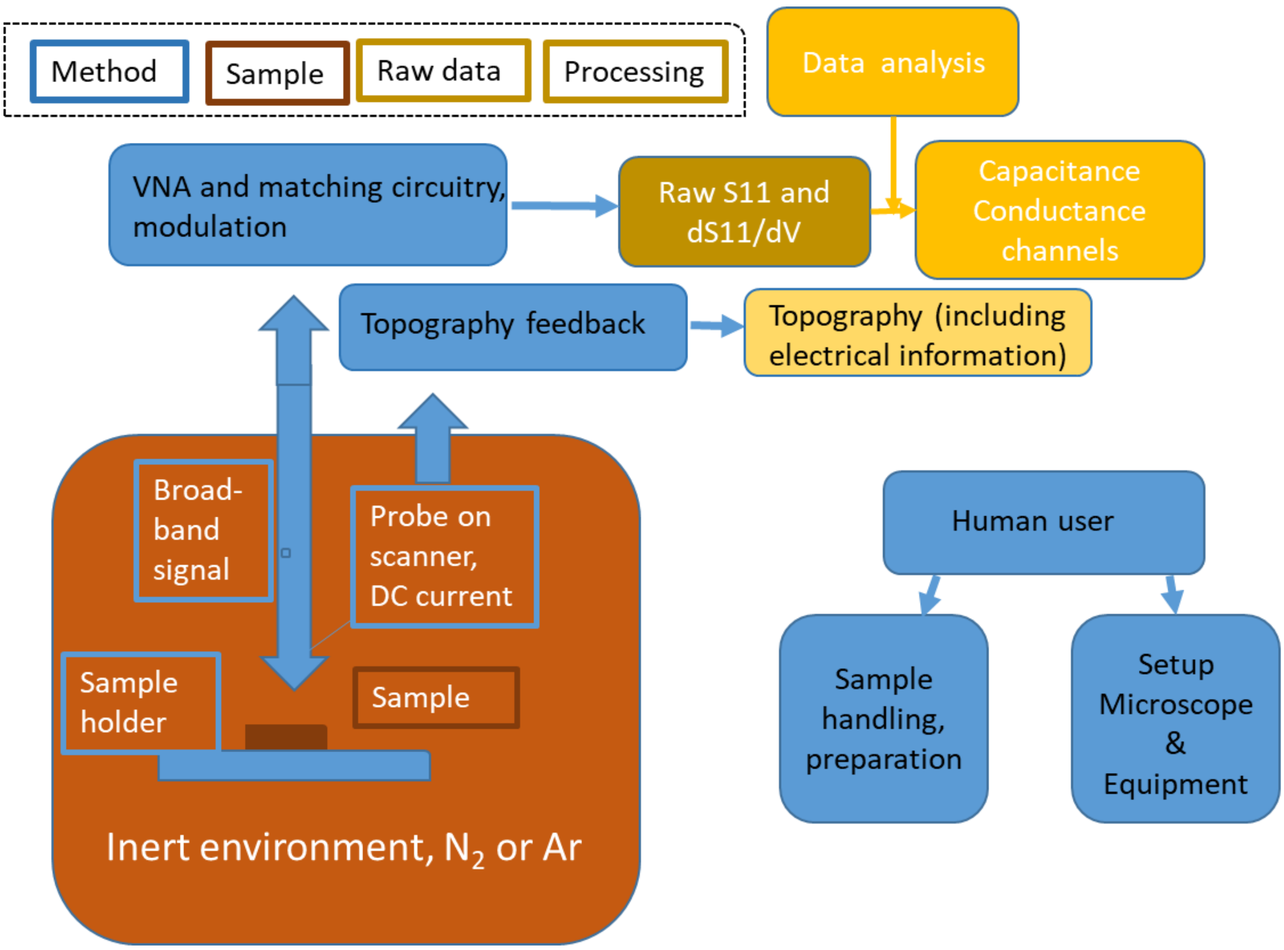
Used in NanoBat (H2020)

Overview of the Characterisation

1	Sample	Open and handle Li-Ion battery half-cells electrodes and electrolyte in inert, dry atmosphere (argon)
2	Chain of methods Method	bb-IM prep 1: all electronics is switched on and checked
		bb-IM prep 2: probe is prepared and isolated freshly
		bb-IM prep 3: Selection of all input parameters
		Glovebox prep: Argon filled glovebox is prepared to guarantee dry, inert atmosphere
		Sample prep 1: Half-cell electrode is transferred to the glovebox, unpacked and glued onto the sample holder
		Sample prep 2: Electrolyte is applied onto the electrode, reference and counter electrode are mounted and checked
		Sample prep 3: Sample is placed inside the microscope
		Approach: Tip is brought in contact with the electrolyte, check connections
		Approach: Tip is approached to the sample
		Calibration: approach curves are used to define the capacitive direction
		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) *A direct link will be added after the publication is available.*
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. Measured impedance data is calibrated with approach curves 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)	Electrodes for Lithium-Ion batteries. Sample dimensions approx. $10x10x1mm^3$. The electrode is glued onto a sample holder on the protective atmosphere in the 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. Low noise and vibrations.
1.5	Material	Main properties of the material under investigation: conductive and dielectric properties, electrochemical reactions, dependency on the electrochemical potential.

	2. Methods	
2.1	Sample/probe physics of interaction	A modulated broad frequency (1kHz-10GHz) 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 DC current. Vector Network Analyzer to generate and measure the radio wave. Potentiostat to apply electrochemical potential. Additional signal generator for modulation.
2.4	Calibration	Capacitance calibration: Retraction curve on bare metallic electrode and electrochemical voltage sweeps.
2.5	Probe	Etched or cut PT/IR probes isolated by wax
2.6	Detector	The vector network analyzer (VNA) measures the amplitude and phase of the S11 parameter. Lock-in amplifier measures modulation of the signal
2.7	Signal	Complex S11 parameter, derivative of S11 with respect to modulation voltage Z Piezo sensor voltage
2.8	Time lapse	Prep: Sample preparation and machine setup 1- 4 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, dS11/dV amplitude and phase in a wide frequency range

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) and modulated signal
3.2	Data acquisition rate	up to 256 px/s

4. Data Processing		
4.1	Main data filtering processes	The S11 filtering is defined by the intermediate frequency bandwidth (IFBW). Modulated signals (dS11/dV) are filtered according to low-pass filter of lock-in.
4.2	Main data analysis procedures	Calibrated S11 and dS11/dV is obtained by approach curves and voltage sweeps.
4.3	Main processed channels	Topography, amplitude and phase of S11 and dS11/dV
4.4	Data processing through calibrations	The calibrated topography is obtained by mapping the piezo sensor voltage to the corresponding piezo expansion. Approach curves and voltage sweeps are used to calibrate S11 and dS11/dV data as detailed in 2.4.
4.5	Properties (elaborated data)	Changes of dielectric and conductive sample properties, topography, charge transfer reactions of various frequencies