TITLE: Data acquisition with a Scanning Microwave Microscope in dry and liquid environment

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SHORT DESCRIPTION: This document's intention is to provide a generic and instrument-unspecific guideline to help in the process of acquiring reliable and reproducible data with the Scanning Microwave Microscope (SMM) under dry conditions and in liquid. This document applies as well to "Scanning Microwave Impedance Microscopy" or "Microwave Impedance Microscopy". Areas covered are sample and microscope handling, data storage and a few quick reminders on data analysis. Extensive advice on data analysis is beyond the document's scope.

ABBREVIATIONS / TERMINOLOGY:

SMM – Scanning Microwave Microscope
VNA – Vector Network Analyzer
S11 – Scattering parameter
SPM – Scanning Probe Microscope
STM – Scanning Tunneling Microscope

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In general a SMM, or sometimes called "Scanning Microwave Impedance Microscope " or "Microwave Impedance Microscope", consists of a Scanning Probe Microscope (SPM), which is interfaced with devices to measure vector microwave reflection and/or transmission, like a VNA [1,2]. Therefore, it is inevitable to respect requirements of the mostly mechanically sensitive scanning probe part of the SMM and the sensitive high frequency components.
The following is a collection of advice for good practice to ensure stable and reliable operation of a SMM to obtain high quality data. The content is kept generic and avoids too much detail, which is instrument or lab specific. The document instead focusses on general good practice, which can be followed in any lab operating a SMM. For operation in dry environment, text passages on liquid environments can simply be skipped.
There are three sections in the following. The first one deals with the lab and setting up a SMM in general as well as sample and data storage. This serves as the foundation and should in the best case only to be followed once, but reviewed on a regular basis. The second part provides advice on the actual measurement process, which is to be followed in day-to-day activities. The last part provides some general advices on the analysis of SMM data.

GENERAL:

Performing experiments with a SMM might expose the operator to laser light, high voltage, sharp objects, chemicals and other hazards thus it is important to ensure proper training of the SMM operator according to local safety regulations and device specificities. This includes familiarity with available handbooks, lab regulations or other available documentation specific to local premises and instruments.

Ensure stable lab climate (temperature, humidity) to enable stable operation of the SMM with minimum mechanical, thermal, electrical drift and liquid evaporation. Avoid opening windows, direct sunlight on the experiment, sources of electromagnetic radiation, etc. In the best case, humidity, temperature and pressure are logged to allow tracing back implausible SMM measurements and check possible influences of one of these parameters. Be aware of the liquids' toxicity and possible chemical reactions by dipping SMM probes into them.

Ensure calm, vibration free environment for the SPM and VNA including the interfacing cables - especially while experiments are conducted. The SMM should be placed in a calm position in the lab, away from noise sources, ventilation and passersby. In the best scenario, the SMM should be shielded from external changes in electromagnetic field e.g. by putting it in a Faraday cage. It should be placed in an acoustic case and on a vibration isolation mount like an optical table or other damping platform. To reach maximum control on environmental variables, or if required for chemical reasons, the SMM can be placed in a glovebox hosting a controlled nitrogen-, or argon-environment. Thereby, hazards, degradation of the liquid or sample, and condensation of water on sample and probe are minimized.

Ensure proper connection of coaxial cables from VNA to the SMM probe. Clean connectors, use a torque wrench and ensure a stable position of all the cables and respecting their specific minimal bending radius. Moving cables can lead to significant change in S11 (especially the phase). Keep cables as short as possible to reduce losses. Make sure cables fit to the frequency range used in experiments.

For measurements with potentiostatic control, ensure proper connection and consistent set-up of counter-, reference-, and working-electrodes according to the lab standards.

SMM measurements under dry conditions can be performed with probes, which are not fully shielded until the very tip, e.g. STM probes. In liquid only shielded probes, where the conductor is protected, can be used, e.g. coaxial probes.

Samples and SMM probes should be stored in a clean, dry, dust free desiccator at stable temperature. Even better options for storage are nitrogen dry boxes or vacuum chambers.

Ensure proper and unambiguous sample labelling to avoid mixing up during storage and experiment.

When handling samples and sensitive parts of the SMM it is advisable to wear nitrile gloves and use tweezers where appropriate. For sample manipulation in "dice" configuration, plastic tweezers are recommended. In contact with chemical liquids, stainless steel is generally appropriate, though be aware of chemical reactions depending on the specific substances. Avoid electrostatic discharge by proper grounding of the operator. In any case, while handling SMM probes any mechanical contact to the tip has to be avoided to prevent both mechanical and electrical damages.

Work according to local IT policies and ensure safe data storage with regular backups. Best to avoid local hard drive and use a server storage.

Keep raw data and post-processed data separate and avoid overwriting raw data during analysis treatment.

MEASUREMENT:

Make sure all relevant devices are switched on. To ensure stable operation the VNA should be switched on, with source power off, for at least a day before measurements start.

Confirm the probe's status and make sure it is still in proper shape for measurements. Perform visual inspection of the sample before measurement and check for dust, scratches, etc. Depending on availability and suitability (some samples can be damaged by being exposed to high-energy electron beam), this can be done using an electron or optical microscope. Before starting measurements, an instrument specific warm up period should be respected.

Make sure to log all relevant metadata (sample, experimental settings, environmental conditions, etc.). An example of a comprehensive set of metadata for SMM and other microwave techniques can be found on DOI 10.5281/zenodo.5524498.

Choose suitable microwave frequency for the experiments. The setting parameters as input RF power, operation frequency, intermediate frequency bandwidth (IFBW), scanning area, scan speed, scan direction, number of points/pixels are chosen depending on the material physical and geometrical properties as well as the characteristic response of the microwave electronics.

For measurements performed in liquid, ensure that the liquid's volume reduction through evaporation does not influence the measurement. This concerns in particular the influence of an ionic liquid's concentration and, depending on the apparatus, the tip-sample distance control. Start the measurement with a reasonably maximal amount of liquid and keep the measurement time as short as needed to avoid changing conditions.

Strictly minimize vibration and (electromagnetic) noise inside the glovebox during the measurement by switching off or unplugging unnecessary instruments, and by reducing the glovebox's ventilation as much as possible.

Before doing SMM measurements on a sample of interest, test the electrical sensitivity of the SMM probe by scanning on well-defined samples or calibration kits for SMM. Currently, there are commercial kits, i.e. capacitive kits consisting of different dielectric (typ. SiO_2) terraces [3]

or resistive/inductive and capacitive gold structures on silicon nitride membranes provided by METAS [4,5].

It is highly recommended to calibrate the SMM to get reliable and comparable quantitative results, which further can be traced back to SI units.

Save data according to local naming convention using unique names. Useful schemes could include sample ID, microwave frequency, scan parameters (range, pixels), timestamp.

After experiments, the SMM should be brought back to standard conditions agreed on in the lab and samples have to be removed and stored safely. If measurements have been performed in liquid, samples, probes and further components should be cleaned by rinsing with appropriate solutions from residuals; in lab environment these are e.g. acetone, isopropanol, and deionized water; in dry glovebox environment e.g. the used solvent, though in no case water or aqueous solutions! Liquids should be disposed safely according to local safety regulations.

ANALYSIS :

One or more of the following types of data are usually generated during SMM measurements:

- Uncalibrated S11 in real/imaginary or abs/angle units,
- Topography, resonator's phase, dissipation, excitation amplitude.

These quantities can be stored either for points, line scans or complete 2D images or in rare cases even 3D images.

Cross-check topography and S11 to distinguish topography-induced S11 signals (unwanted) and those of purely material-related origin (wanted).

Even though Phase (S11) and Magnitude (S11) reproduce very often, it is necessary to carefully check both for unique features.

Further analysis according to common practice.

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REFERENCES:

[1]: G. Gramse, M. Kasper, L. Fumagalli, G. Gomila, P. Hinterdorfer, and F. Kienberger, “Calibrated complex impedance and permittivity measurements with scanning microwave microscopy”, Nanotechnology **25**, 145703 (2014).

[2]: Arne Buchter et al., "Scanning microwave microscopy applied to semiconducting GaAs structures", Rev. Sci. Instrum. **89**, 023704 (2018).

[3]: José Moran-Meza, Alexandra Delvallee, Djamel Allal and François Piquemal, "A substitution method for nanoscale capacitance calibration using scanning microwave microscopy", Meas. Sci. Technol., 0957-0233, 2020.

[4]: T. Le Quang, D. Vasyukov, J. Hoffmann, A. Buchter, M. Zeier, "Fabrication and Measurements of Inductive Devices for Scanning Microwave Microscopy", IEEE-Nano, 429-432, 2019.

[5]: T. Le Quang, et al., "Advanced calibration kit for scanning microwave microscope: Design, fabrication and measurement", Rev. Sci. Instrum., 92, 023705, 2021.