TITLE: Data acquisition with a Scanning Microwave Microscope


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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.  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


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In general a SMM, or sometimes be called "Scanning Microwave Impedance Microscopy " or "Microwave Impedance Microscopy", consists of a Scanning Probe Microscope (SPM) which is interfaced with devices to measure vector microwave reflection and/or transmission, like a VNA. 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.

There are three sections in the following. The first one deals with the lab and setting up a SMM in general and sample and data storage. This serves as the foundation and should in the best case only needed 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, and electrical drift. 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.

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 the SMM can be placed in a glovebox which hosts a controlled nitrogen-environment and thus also avoids any condensation or water layers on sample and probe.

Ensure proper connection of coaxial cables from VNA to the SMM probe. Clean connectors, use 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

In the best case, samples and SMM tips should be stored in a clean, dry, dust free desiccator at stable temperature. There are other and better options for storage such as: Nitrogen dry box or vacuum chamber.

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. Plastic tweezers are appropriated for samples manipulations in "dice" configuration. Where necessary avoid electrostatic discharge by proper grounding of the operator. Moreover, while handling SMM tips, it is recommended to

avoid any mechanical contact to the end of the tip, which can cause 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 make sure to not overwrite 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 tip 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 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.2591367.

Choose suitable microwave frequency for the experiments. 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.

Before doing SMM measurements on a sample of interest, test the electrical sensitivity of the SMM probe by scanning on well-defined samples, calibration kits for SMM. Currently, there are commercial kits, i.e. capacitive kits provided by Prime Nano consisting of different dielectric (typ. $SiO_2$) terraces or resistive/inductive and capacitive gold structures on silicon nitride membranes provided by METAS.

It is highly recommended to calibrate the SMM to ensure the traceability of measurements to the SI, *i.e.* to get reliable and comparable quantitative results.

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 sample is to be unmounted and stored safely.


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

- Uncalibrated S11 in real/imaginary or abs/angle

- Topography, phase, dissipation, excitation amplitude

These quantities can be either stored 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 a must to carefully check both for unique features.

Further analysis according to common practice.