Guidelines for Terahertz microprobe-based inspection of thin-film conductors

1 Terahertz Near-field Transmission Measurements

Terahertz near-field inspection is a versatile measurement tool to determine electrical properties of conductive thin-films. Here we focus on the description of THz transmission measurements at graphene layers and the relevant sample properties for such measurements. The information shall help to select appropriate samples for THz transmission measurements.

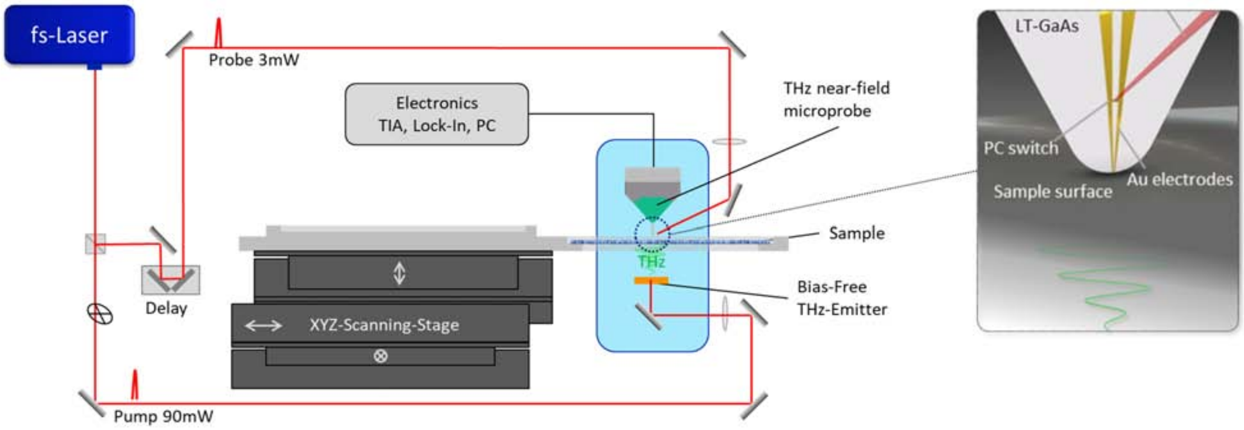

Figure 1.1: Scheme of the Terahertz near-field setup.

1.1 Graphene Sample Properties for THz Inspection

1.1.1 Requirements and parameters of interest:

The electrical properties of Graphene can be determined from Terahertz (THz) measurements. To obtain the required raw data from measurements we need a reference sample of substrate only and a sample consisting of an identical substrate plus graphene on top. By comparing the Terahertz transmission through both samples we can calculate the graphene charge carrier properties from the THz transmission measurements.
In general a large variety of conductive thin-films on different substrates can be inspected. But the quality of the results can be strongly improved, if certain specifications are fulfilled. In the following the sample properties are described that have an influence on the quality and accuracy of the derived graphene sheet resistance (and mobility) data from the THz transmission measurements.

- Sheet conductivity of the (graphene) layer of interest:
  If the layer is too conductive, no THz radiation will be transmitted and can be measured. If the layer is not conductive enough, no absorption within the layer occurs and no signal changes can be measured. Graphene sheets with a conductivity of typically some $0.1\,\mathrm{mS}/\square - 10\,\mathrm{mS}/\square$ are usually well suited for inspection.

- Reference area:
  To obtain quantitative sheet conductivity results for a layer of interest, a reference area is needed, that does not carry the conductive sample layer. The reference substrate should be identical to the sample area substrate, just without the conductive layer of interest. An ideal reference area would be a position without deposited graphene on the same wafer, so it has undergone the same process steps and it is ensured, the substrate is identical for reference and measurement area.
  But also a complete second substrate wafer is suitable as reference. It should be identical to the graphene sample substrate (same specifications, same wafer batch, ideally neighbored). Minimum size of a reference area should be at least some mm in each direction.
  (For conductivity estimations it is also possible to use a sample area with known sheet conductivity for referencing, instead of using a completely layer-free reference area. In this case the known conductivity in those areas can serve as reference value input to calculate the conductivity for the other sample regions.)

- Homogeneous substrate:
  The substrate material needs to be homogeneous in terms of thickness, conductivity, THz transparency and refractive index. As the transmitted THz radiation passes all substrate and sample layers, any inhomogeneity will be detected and might be mis-interpreted as conductivity variation within the graphene.
  Also if multiple samples are to be directly compared, this comparison will be most accurate, if all samples are based on identical substrate.

- Multi-layer stacks:
  The same argument applies to stacked layers. As long as all layers are sufficient THz transparent and homogeneous except for the sample layer of interest, multi-layer stacks are no general problem.

- Substrate conductivity and THz transparency:
  The more THz light can pass the substrate and interact with the sample layer before being detected, the better the signal quality and therefore the measurement result is. Thus substrates should be as THz transparent as possible. Examples for well suitable substrate materials are sapphire, high-resistivity silicon, crystalline quartz. Samples based on moderately doped semiconductors can often still be inspected (with reduced quality). Strongly doped semiconductors or metallic layers within the substrate prevent THz transmission.

- Constant substrate thickness:
  Thickness variation for conductive substrates will always come along with inhomogeneous THz absorption within the substrate and should be completely avoided. For substrate with high THz transparency (low conductivity) thickness variations smaller than $1\,\mu$m can usually be neglected. Larger thickness variation of up to some $10\,\mu$m can often be compensated by spending additional measurement time on that sample. THz transparent samples with strong thickness variations can still be measured, but at some point the extraction of quantitative conductivity information becomes difficult.

- Substrate curvature:
  Samples based on rigid, flat substrates like wafers are most convenient for THz near-field inspection. But also flexible materials like PET or other dielectrics can be used as substrate material. For high-resolution measurements, the distance between near-field detector and sample needs to be held constant, but the measurement system can compensate small height variations and can be used for the inspection of bended samples on flexible substrate. Samples with strong curvature or even varying orientation of the sample area can not be inspected with high resolution and accuracy.

- Sample Size:
  Sample- and scanning area size are mainly limited by the need to mount and move the samples in the setup. Samples larger than $1\,\mathrm{cm}^2$ and smaller than $140x120\,\mathrm{mm}^2$ can be easily measured without stitching. For 6" wafers most of the sample area can be directly measured. Stitching might become necessary for larger samples and usually solutions can be found to measure very small samples.

- Measurement resolution and measurement duration:
  The maximum scanning resolution is some few μm. Very accurate measurements with resolutions better than some $10\,\mu$m require additional effort and should only be used for specific sample areas having known small features. For Wafer-scale inspection a first scan with some $100\,\mu$m resolution assisted by higher resolution measurements of interesting areas is usually the best solution.
  Measurement time scales quadratically with the measurement resolution. For conductivity measurements scanning durations of some $3 - 30\,\mathrm{ms/px}$ are typical, while for high-quality mobility inspection theses time can increase to some seconds/px.

1.1.2 Parameter Values

The following table summarizes the various parameters that have an influence on THz transmission based conductivity (and mobility) measurements. It rather serves as an orientation and is not an exact definition.

Parameters and Grouped Values			
	Optimal	Acceptable	Difficult
Graphene R_{sh}	$5 - 500\,\Omega/\square$	$0.5 - 5000\,\Omega/\square$	$< 0.1\,\Omega/\square$ $> 10000\,\Omega/\square$
Substrate Material	Sapphire Al_2O_3, Crystalline Quartz SiO_2, Highly resistive ($\rho > 100\,\Omega$cm) semiconductors	Flexible Dielectrics, Lightly doped Semiconductors	Paper, Strongly Doped or metalized Substrates
Substrate Curvature and stiffness	Flat and rigid	Flexible and slightly bended	Strongly curved or 3-dimensional
Substrate Thickness variation	$< 1\,\mu$m	$< 20\,\mu$m	$> 100\,\mu$m
Spatial Resolution	$25 - 250\,\mu$m	$10 - 10000\,\mu$m	$< 5\,\mu$m
Sample Size (longer edge)	$20 - 100$ mm	$5 - 150$ mm	< 1 mm > 300 mm
Measurement Points	100 Px – 100 kPx	< 1 MPx	> 1 MPx

Reliable quantitative measurement results require a good THz transmission signal-to-noise ratio. To obtain accurate mobility values for charge carriers in graphene, most sample parameters should be "optimal". The extraction of graphene sheet conductivity or resistance is more robust than mobility measurements and usually reliable values can already be obtained for samples fulfilling "acceptable" conditions. If your sample parameters are outside these specifications it might still be possible to get at least qualitative results.

1.1.3 Exemplary Conditions

A scheme of a sample with perfect conditions is shown in figure 1.2. With such a sample design even unexpected inhomogeneities of the substrate material would be detected during the THz measurement and will not be misinterpreted as variations within the sample material itself. But this is just an example and other measurement settings are possible as well, as depicted in figure 1.3. Not all substrate and sample properties need to fulfill perfect conditions to allow quantitative THz measurement results. Some substrate inhomogeneities can be compensated by additional measurement efforts, when their existence is known.
Unless the substrate material is completely THz opaque or the sample material shows no relevant THz absorption, qualitative inspection can usually be done and the results can be interpreted in terms of sheet conductivity.

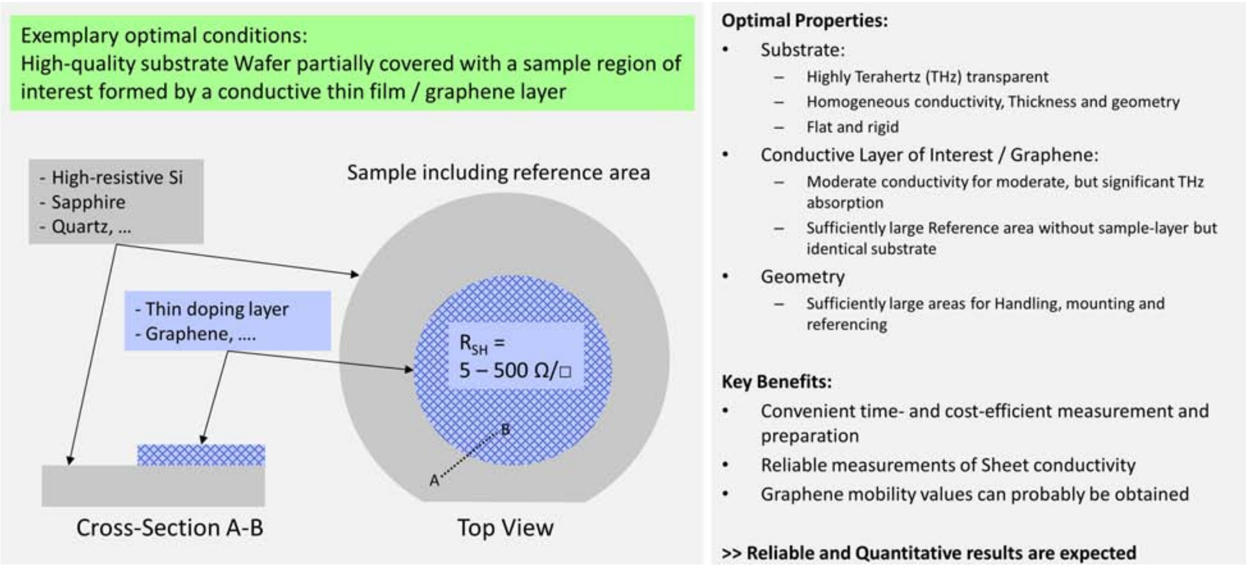

Figure 1.2: Scheme of an exemplary sample with perfect settings for THz transmission measurements.

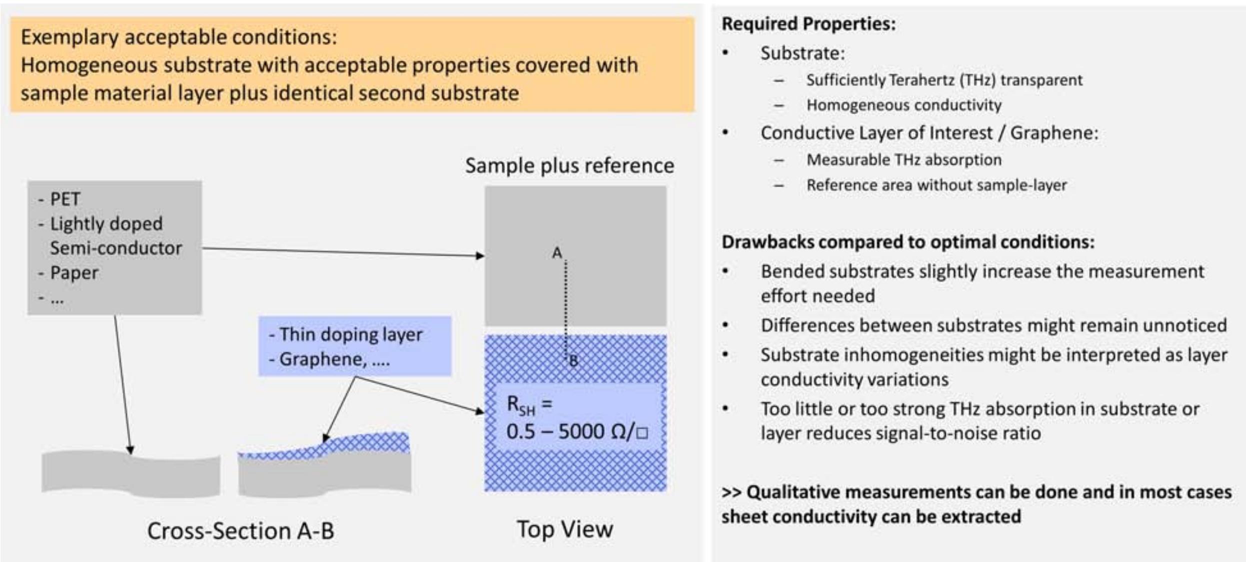

Figure 1.3: Scheme of a sample with not perfect, but still acceptable properties.

1.1.4 Exemplary Measurement Results

Exemplary measurement results are shown in 1.4. The 4" graphene sample is completely supported by a 6" silicon wafer, leaving enough space to select reference areas and for safe handling of the sample. The graphene layer shows conductivity variation on the wafer-scale, as well as small defects that were inspected with another high-resolution mapping. Graphene-typical defects like cracks and wrinkles especially at the graphene borders can be identified.
The substrate wafer is sufficiently THz transparent to allow reliable sheet conductivity measurements. The mobility of the charge carriers in the for this specific graphene sample could be extracted only with a large uncertainty.

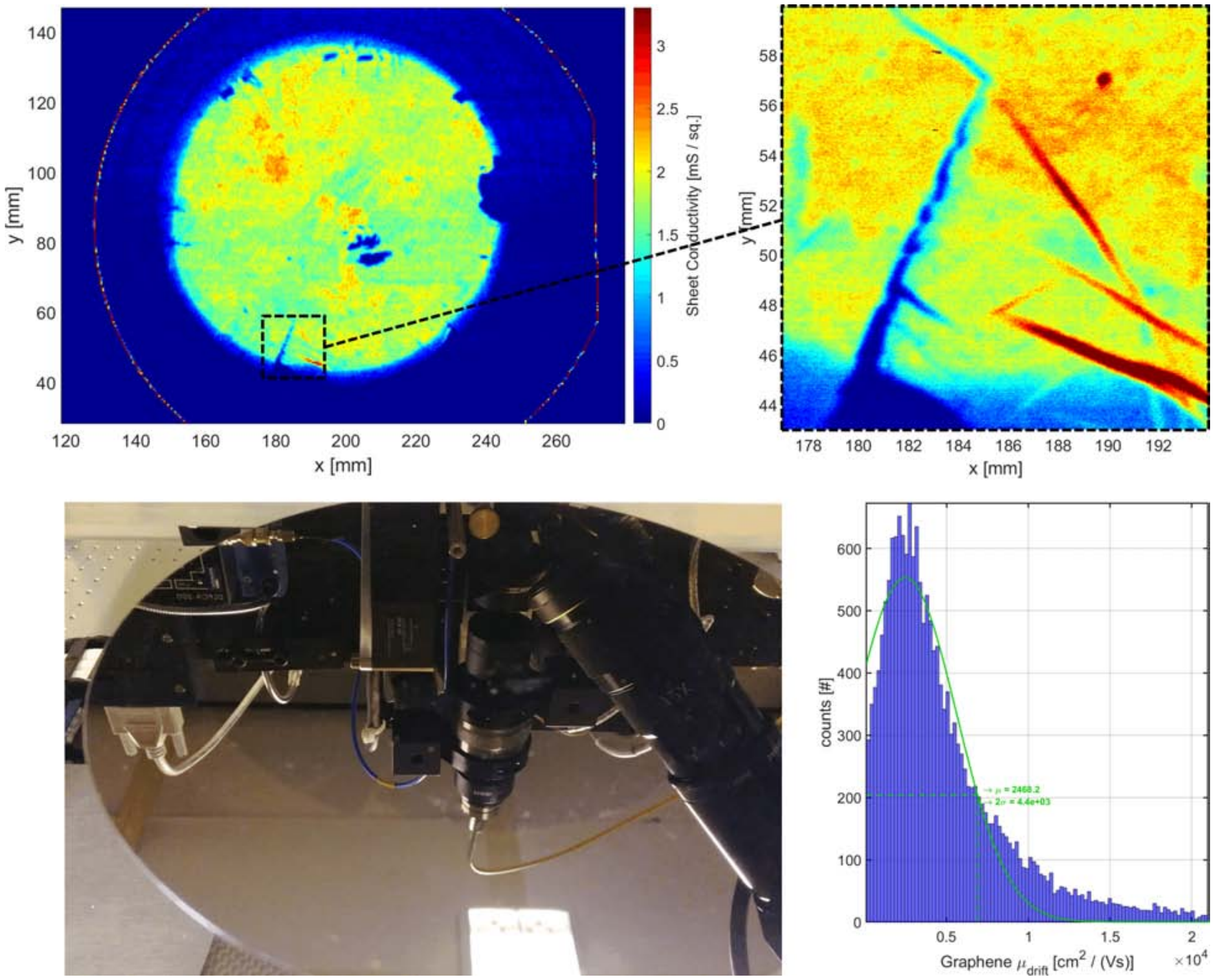

Figure 1.4: Top Left: Exemplary THz Transmission measurement results from a 4" graphene layer on a 6" silicon wafer. Top Right: Higher Resolution mapping of a smaller region showing conductivity variations due to cracks and wrinkles in the graphene layer. Bottom left: Photograph of the sample in the measurement setup. Bottom Left: Mobility Histogram.

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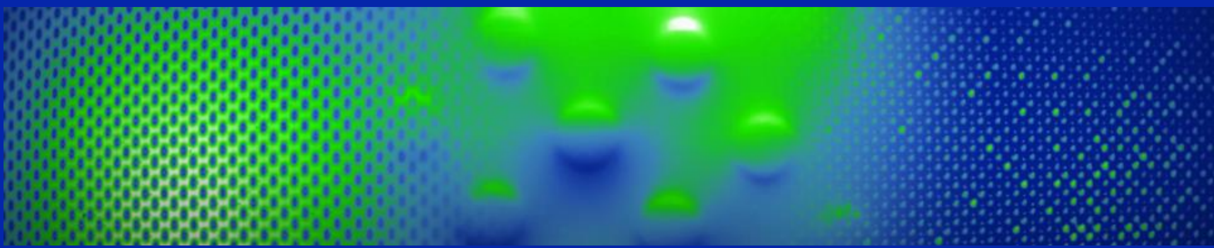

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