Conductive AFM (CAFM) module for NanoWizard® AFM

AFM is a versatile tool not only for the measurement of sample topography but also for the characterization of mechanical and electrical properties on the nanometer scale. For many materials such as organic semiconductors, graphene, CNTs or nano-spun fibers, biosensors and lithium battery components, the characterization of the electrical properties plays a major role to increase the performance of next-generation devices. The basic requirements to derive the electrical properties of a sample by AFM are a conductive probe and dedicated electronics. Based on this it is possible to measure low-level currents between the probe tip and a sample as the probe is scanned over the sample.

The relation between electrical and mechanical properties on the nanoscale is of great importance as it can have a tremendous influence on the usability and performance of a material in real-world devices. However, deriving real mechanical data instead of contrast only is really a challenge when using conventional AFM imaging modes.

For studying soft and delicate samples conventional conductive AFM is not well suited. The reason is the classic contact mode imaging which typically results in sample damage and tip wear or contamination. In order to solve the problems of conventional AFM imaging modes, JPK has developed the new QI™ imaging mode for all NanoWizard® 3a AFMs. Due to its unique scanning scheme, the QI™ imaging mode eliminates lateral forces while providing precise control of the vertical forces acting between tip and sample. Thus, it allows the non-destructive and intuitive imaging of not only very soft but also sticky, brittle or loosely attached samples. Additionally, the new QI™ Advanced imaging mode provides mechanical information which is derived from well-established contact mechanical models through the analysis of true force curves captured during imaging.

Fig. 1: Conductive AFM module from JPK now used in combination with QI™ Advanced

Fig. 2: The CAFM module with environmental control supports experiments under defined environmental conditions like oxygen-free atmosphere or controlled humidity.

Fig. 3: The sample holder for opaque samples allows the bias voltage to be guided to the sample. A holder for optically transparent samples is also available.
Setup Description

As seen in Figs 1 and 2 the CAFM modules from JPK are specially designed cantilever holders. The standard version is made for experiments under ambient conditions. The version with environmental control has a closed cell that covers a volume of 140 µL approx. The transparent parts of the JPK CAFM environmental cell are made from polycarbonate.

In both cases the conductive AFM module is equipped with a circuit board that needs to be close to the cantilever for reliable low current amplification. All standard CAFM cantilevers are compatible. The chip and the glass holder are held together with a mechanical clip that also collects the current from the tip.

Two different sample holders are available to support opaque (Fig. 3) or transparent samples. The sample size is variable. Sample diameters can go up to several cm. High NA objectives can be used to study the sample optically with inverted or upright microscopes.

In combination with the JPK Conductive AFM module (see Fig. 1), the QI™ Advanced imaging mode is able to provide electrical information for a sample on the nanometer scale, while maintaining the highest topographic resolution. Besides being easy to use, another advantage of the force curve based approach is the capability to further analyze the conductive data in more detail at every pixel, which is not possible with data from conventional conductive AFM measurements.

The measurable current ranges from sub-pA levels up to several µAs. By an advanced treatment of the captured data, like a local averaging in the simplest case, the resolution limit can be further extended down to the fA range.

Fig. 4: A conductive polymer foil (conductive particles, embedded in non-conductive polymer matrix) was imaged using the JPK C-AFM module. A constant bias was applied between the conductive tip (Pt coating) and the back electrode under the sample. Conductive particles (blue / red), embedded into the non-conductive polymer matrix (white) are clearly visualized in the current image.
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The broad compatibility to other accessories for the NanoWizard® AFM series including the temperature and environmental control solutions as well as excellent optics enables the most sophisticated AFM experiments.

Fields of application

The CAFM modules are made for middle to low current recordings, down to the sub-pA range. Conducting volume samples, and conductive films, as well as semiconductors can be used as samples for a CAFM study. Recording low currents during imaging is a standard application, but currents can also be recorded during local oxidation experiments when the conductive cantilever is used as a manipulating tool. Force spectroscopy applications also can be applied to CAFM experiments when current flow is of interest depending on the force that is applied to the sample. Constant voltages or voltage ramps of different shape between +/- 10 V and down to millivolts in any case can be applied to the sample during an experiment.

The new QI™ Advanced imaging mode for NanoWizard® 3a for conductive measurements is especially of benefit, but not limited to, the mapping of conductive properties on soft and brittle samples. Together with dedicated solutions for environmental and temperature control as well as the integration with optical techniques, a broad range of applications becomes available. Key examples include organic semiconductors such as organic photovoltaics, biosensors, displays and battery components. The investigation of the properties of graphene or CNTs is growing in importance.

Also for the measurement of rather stiff samples, the QI™ Advanced imaging mode is advantageous, as it eliminates lateral forces and therefore tip wear. Tip wear is of major concern when performing conductive AFM due to potential abrasion of the conductive coating of the cantilever.

Fig. 5: Overlay of 3D topography and current image: Conductivity can only be observed on the grainy particles, not on the polymer matrix.

Fig. 6: Conductive QI™ measurement of a graphene flake on a conductive Ir/Au substrate. (a) 3D-topography (scan range: 1.5 x 1.5 µm²). (b) Representation of 3D-topography overlaid with color scale of current image. As can be seen the graphene flake is non-conducting perpendicular to the substrate. (c) A typical force curve as derived during QI™ imaging presenting vertical deflection (red).
Typical applications include:

- (Organic) semiconductors and compounds
- Graphene
- Nanotubes
- Dielectric and ferroelectric thin films
- Nanoparticles and quantum dots
- Conductive polymers and polymer coatings
- Light emitting polymers
- Biosensors and MEMS
- Lithium ion battery components
- Local oxidation

**Fig. 7:** Local oxidation on Si, done with a conductive cantilever used for manipulation. Voltage ramps between -4 and -10 V have been applied during the writing. Current has been recorded during the writing.

**Fig. 8:** A I/V curve, recorded on a graphite-clay blend.

**Fig. 9:** The JPK logo, written by local oxidation of an OTS monolayer on Si. Phase image. Sample courtesy of Prof. J. Sagiv, Weizmann Institute, Israel.

**Conclusion**

The JPK Conductive AFM module is perfect for low noise and high performance current measurements even in combination with a controlled environment. A choice of different modules with different current ranges gives the user maximum flexibility.

Apart from standard applications for Conductive AFM in material characterization new classes of materials being developed for example for green and low-cost electronics set a new benchmark for the characterization of electrical properties by AFM. Their mechanical properties make it impossible to apply conventional modes of conductive AFM imaging. With the new QI™ Advanced imaging mode coming with the NanoWizard® 3a, JPK has developed a solution for the imaging of these kinds of samples. Aside from providing electrical data, this new imaging mode also allows the correlation of electrical and real mechanical data.
Specifications

- High-performance conductive AFM experiments
- Also recording I/V curves or for local oxidation
- Bias voltage range ± 10 V for all modules
- Different current ranges available
  - The low current version with a gain $1\times10^8$ [V/A] up to ± 100 nA with a noise limit 0.5 pA RMS
  - The high current version with a gain $1\times10^5$ [V/A] up to ± 100 µA with a noise limit of 80 pA RMS
  - Other current ranges on request
- Sample holder for opaque samples (standard)
- Sample holder for transparent samples works with all inverted optical microscopes
- Optional conductive module version with closed sample volume for experiments in defined atmosphere with a volume of 140 µL
- The Conductive AFM module is compatible with all NanoWizard® AFM’s
- In combination with QI™ Advanced mode and NanoWizard® 3a mechanical and electrical data can be correlated on the nanoscale in a single scan
- With QI™ Advanced high resolution and low noise current mapping on fragile, brittle and loosely attached samples, virtually no tip wear due to eliminated lateral forces
- Parameters such as start and end voltage of the ramp, ramp-direction or rate can be easily chosen in the user interface. The software can either record a single spectrum or average over multiple spectra. With the ExperimentPlanner™ software module the user has the absolute freedom of designing individual measurement procedures
- Compatible with JPK heating/cooling stages (see JPK accessories handbook)
- For experiments under controlled gas atmosphere the setup is compatible with the JPK glove box solution (see product note)