Introduction

Controlling the environment of scientific experiments has always been good laboratory practice for all researchers. It is a key ingredient to ensure the reproducibility and repeatability of experiments as well as for achieving meaningful results. Parameters to be controlled can range from temperature over electromagnetic fields to temperature and composition of the gas atmosphere of the experiment. Controlling the sample’s gaseous environment becomes even more critical and essential for some new promising technologies for saving, storing, or producing electrical energy: Lithium ion batteries and organic solar cells. The components of these technologies are sensitive to chemical modification and particularly to oxygen exposure. Thus devices built on these technologies generally have to be encapsulated and sealed very well.

Keysight Technologies, Inc. has a long tradition in providing Scanning Probe Microscopy (SPM) systems which allow running experiments in controlled sample environments\textsuperscript{[1-3]} without compromising on SPM performance. More recently the new technologies demanded also for systems allowing the investigation as well as the sample preparation being carried out in a completely controlled gaseous environment. To meet these requirements Keysight Technologies has teamed with MBraun Inert Gas Systems and Accurion to integrate high resolution SPM into Laboratory Glove boxes enabling a seamless workflow from sample preparation to high resolution investigation under controlled atmospheres.

This Application Note reports investigations involving high resolution SPM experiments with Keysight Technologies SPMs working in a glove box environment without compromising the SPM performance. More recently the new technologies demanded also for systems allowing the investigation as well as the sample preparation being carried out in a completely controlled gaseous environment. To meet these requirements Keysight Technologies has teamed with MBraun Inert Gas Systems and Accurion to integrate high resolution SPM into Laboratory Glove boxes enabling a seamless workflow from sample preparation to high resolution investigation under controlled atmospheres.

Inert Gas Environment

The SPM experiments were carried out in an up-to-date MBraun Inert Gas System glove box with a reinforced support to provide a solid platform for the SPM operation. A three glove solution is indicated for the large sample AFM 5600. The compact 5500 AFM can be operated in a two glove solution. Two load locks allow the exchange of samples and accessories without breaking the inert gas atmosphere. The inert gas in the system can be Argon or Nitrogen and is cycled through a regenerator system at a rate of up to 88 cubic meters per hour. The gas flow necessary for the continuous purification of the gas was designed to have minimum impact on the SPM operation. Fans and electronics for the operation of the system are located in a separate rack which is connected to the glove box via soft flexible tubing. The control system measures the content of oxygen and water in the return line. The levels were kept below the 0.1 ppm detection limit throughout the operation. The control system allows reducing flow rate which minimizes the noise input to the SPM system. A detailed description of the system can be found under\textsuperscript{[4]}. The system was operated with an Argon atmosphere.

All SPM data reported here were acquired in oxygen free and humidity free Argon atmosphere, i.e. oxygen and humidity levels were measured to be less than 0.1 ppm. Figure 2 presents an example of ultimate resolution of the Keysight 5500 STM showing atomic resolution of the HOPG graphite surface.

Figure 1. Photograph of the AFM 5600 supported by the active damping inside the glove box. The inset shows the operation panel of the glove box indicating the measured impurity concentration at levels below 0.1 ppm.

Figure 2. Example of ultimate resolution of the Keysight 5500 STM presented as 3D showing atomic resolution of the HOPG graphite surface. The glove box gas flow was reduced to 20%.
Antivibration System

The Keysight Technologies 5600LS and 5500 SPM units were supported by Accurion GmbH anti vibration systems. The systems consist of compact damping modules inside the inert gas environment and an external control unit. The damping modules comprise the sensors monitoring the vibration levels and the electromechanical actuators which compensate the incoming vibrations. Vibration levels on top of the platform are reduced by a factor of -40 dB beyond frequencies of 10 Hz.[5]

Scanning Probe Microscopes

Two Keysight Technologies SPM systems were used in the controlled environment: The 5600 large stage system and the 5500 AFM/STM.

AFM 5600

The 5600LS platform features a 200 mm x 200 mm motorized sample stage which is fully addressable over the full range with a repositioning accuracy of 500 nm. The system’s Multipurpose Scanners allow for a wide range of SPM modes for topographic, mechanical, electrical, magnetic, electro-chemical properties and more. It is suitable for operation on dry surfaces or in liquids. The modular electronics provides multiple lock in amplifiers and built in bi-potentiostats. Dedicated STM scanners are available.

Figure 3(a) shows a 300 nm scan range, high resolution AC mode topography and phase imaging of C60 Alkanes forming ordered layers on an HOPG substrate. The molecules form domains with different orientations of the ordered rows which have a periodicity of 7 nm. The rows of alkanes were clearly resolved in the topography as well as the phase image for compactness only topography is shown here.

AFM 5500

The 5500 high resolution AFM and STM platform features an industry leading environmental small volume control for inert/aggressive atmospheres, which allows to control a small part of the inert gas volume in the sample chamber, e.g. for degradation experiments without contaminating the whole glove box system. The system’s Multipurpose Scanners allow for a wide range of SPM modes for topographic, mechanical, electrical, magnetic, electro-chemical properties and more. It is suitable for operation on dry surfaces or in liquids. The modular electronics provides multiple lock in amplifiers and built in bi-potentiostats. The flexible sample plate concept provides easy to use liquid cells for electrochemical environments, heating and cooling plates, lateral magnetic field generators, and many more.

Figure 3(b) shows a 160 nm scan range, high resolution AC mode imaging of C36 Alkanes forming ordered layers on an HOPG substrate. The molecules form domains with different orientations of the ordered rows which have a periodicity of 4 nm. The rows of alkanes were clearly resolved in the topography as well as the phase image for compactness only phase is shown here.

Figure 4 features a 7 nm scan range, high resolution AFM topography of the atomic resolution slip-stick on a mica sample. The slip stick behavior is resolved in topography as well as in friction. For this experiment the circulation of the gas inside the glove box was switched off completely without a detectable deterioration of the gas purity.
Figure 5 shows an 8 nm scan range, atomic resolution STM topography of an HOPG graphite sample employing a dedicated STM scanner. The gas flow was reduced to 20% of the normal operation.

The high resolution topography examples above demonstrate the successful integration of high resolution SPM systems into high performance inert gas systems without compromising either SPM performance or purity of the gaseous environment. Furthermore the intelligent designs of glove box and SPM systems enable a smooth operation of the system including the easy exchange of SPM tips or mounting of samples. For the 5600LS system this is combined with the fully programmable motorized stage for automatic inspection of different areas of larger samples or for series of experiments on many samples.

References

2. In Situ Electrochemical Measurements Using 7500 AFM, Keysight Technologies Application Brief 5991-3298EN, 2013
5. http://www.halcyonics.de/

AFM Instrumentation from Keysight Technologies

Keysight Technologies offers high precision, modular AFM solutions for research, industry, and education. Exceptional worldwide support is provided by experienced application scientists and technical service personnel. Keysight’s leading-edge R&D laboratories are dedicated to the timely introduction and optimization of innovative, easy-to-use AFM technologies.

www.keysight.com/find/afm

For more information on Keysight Technologies’ products, applications or services, please contact your local Keysight office. The complete list is available at: www.keysight.com/find/contactus