

**Asiaoptics.com – February 2011**

# **Improving Micro-Raman/AFM Systems Imaging Using Negative-Stiffness**

## **Introduction**

Negative-stiffness vibration isolators can easily support the heavy weight of a combined AFM/micro-Raman system and isolate it from low frequency vibrations more effectively than high-performance air tables or active isolation systems.

The need for precise vibration isolation with scanning probe microscopy (SPM) and near-field scanning optical microscopy (NSOM) systems is becoming more critical as resolutions continue to bridge from micro to nano. Whether used in academic labs or commercial facilities SPM and NSOM systems are extremely susceptible to vibrations from the environment. When measuring a very few angstroms or nanometers of displacement an absolutely stable surface must be established for the instrument. Any vibration coupled into the mechanical structure of the instrument will cause vertical and/or horizontal noise and bring about a reduction in the ability to measure high resolution features – the vertical axis being the most sensitive for SPMs but they can also be quite sensitive to vibrations in the horizontal axis.

Traditionally bungee cords and high-performance air tables have been the vibration isolators most used for SPM and NSOM work. The ubiquitous passive-system air tables adequate until a decade ago are now being seriously challenged by the need for more refined imaging requirements. Bench top air systems provide limited isolation vertically and very little isolation horizontally. Also at a disadvantage are the active isolation systems known as electronic force cancellation that use electronics to sense the motion and then put in equal amounts of motion electronically to compensate and cancel out the motion. Active systems are somewhat adequate for applications with lasers and optics as they can start isolating as low as 0.7 Hz but because they run on electricity they can be negatively influenced by problems of electronic dysfunction and power modulations which can interrupt scanning.

Lately the introduction of integrated microscopy systems employing multiple microscopes is enabling more complex optical measurements but these systems are also much heavier and there has been little vibration isolation technology available for such heavy instrumentation. Air tables which have been liberally used for optics applications are not ideal for these nanoscale resolution systems because of their inability to effectively isolate vibrations below 20 Hz. Nor can active systems be used with these newer combination systems because of their inability to handle heavy instrumentation. But now negative-stiffness mechanism (NSM) vibration isolation is quickly becoming the choice for SPM and NSOM systems. This includes applications using atomic force microscopy (AFM) integrated with micro-Raman spectroscopy where negative-stiffness vibration isolation is particularly well adapted. In fact it is the application of negative-stiffness isolation that has enabled AFMs to be truly integrated with micro-Raman into one combined system. Negative-stiffness isolators can handle the heavy weight of the combined AFM/micro-Raman system as well as isolate the equipment from low

frequency vibrations a critical set of factors that high-performance air tables and active systems cannot achieve.

## AFM with Micro-Raman Integrated

The integration of AFM with micro-Raman enables a sizable improvement in data correlation between the two techniques and expanded Raman measurement and resolution capabilities. Micro-Raman is a spectroscopic NSOM technique used in condensed matter physics and chemistry to study vibrational rotational and other low-frequency modes in a system. It relies on scattering of monochromatic light usually from a laser in the visible near infrared or near ultraviolet range. The laser light interacts with phonons or other excitations in the system resulting in the energy of the laser photons being shifted up or down. The shift in energy gives information about the phonon modes in the system. Scanning samples in a micro-Raman system however suffers from several problems. As a sample is scanned even a very flat sample it is hard to keep the distance of the lens to the sample constant. Thus as one goes from pixel to pixel under the lens of a Raman a mixture of sample and air is sampled in the voxel (volumetric picture element) that is illuminated. This causes intensity variations in the Raman that are unrelated to the chemical composition of the sample and are artifactual. This is even more pronounced with rough samples and standard methods of autofocus are simply not accurate enough for a whole host of problems that are being investigated today. Additionally the point spread function which determines the resolution of the Raman image is significantly broader where there are contributions from the out-of-focus light and this reduces resolution.

The atomic force microscope being a very high-resolution type of scanning probe microscope has demonstrated resolution of fractions of a nanometer making it one of the foremost tools for imaging measuring and manipulating matter at the nano-scale. The information is gathered by "feeling" the surface with a mechanical probe. Piezoelectric elements that facilitate tiny but accurate and precise movements on electronic command enable the very precise scanning.

The AFM consists of a micro-scale cantilever with a sharp tip (probe) at its end that is used to scan the specimen surface. The cantilever is typically silicon or silicon nitride with a tip radius of curvature on the order of nanometers. When the tip is brought into proximity of a sample surface forces between the tip and the sample lead to a deflection of the cantilever. Resultant characteristics such as mechanical electrostatic magnetic chemical and other forces are then measured by the AFM using typically a laser spot reflected from the top surface of the cantilever into an array of photodiodes.

Most systems employing AFM in concert with Raman perform separately executing either an AFM scan or a Raman scan independently. The recently developed direct integration of Raman spectroscopy with AFM technique however has opened the door to significantly improved technique and sample analyses.

Micro-Raman is a microtechnique but when AFM is added it becomes a nanotechnique. It allows the AFM structural data to be recorded online and improves the resolution of the Raman information when the nanometric feedback of the system adjusts with unprecedented precision the position of each pixel of the sample relative to the lens. Also the small movements of the AFM stage provide oversampling which is a well-known technique for resolution improvement.

One integrated AFM-Raman system developed by Nanonics Imaging Ltd. in association with major Raman manufacturers such as Renishaw plc Horiba JY and others provides simultaneous and very importantly on-line data from both modalities. This advantage addresses critical problems in Raman including resolution and intensity comparisons in Raman images while permitting on-line functional characterization such as thermal conductivity elasticity and adhesion electrical and other properties. It also provides for new avenues of improved resolution including AFM functioning without optical obstruction parallel recording with Raman in a wide variety of scanned probe imaging modalities enabling direct and simultaneous image comparison and analysis and high-resolution Raman mapping.

“Until recently Raman scattering has remained separate and removed from the proliferation of insights that the scanned probe microscopies can give” says Aaron Lewis President of Nanonics Imaging which was the first to see the potential of such integration. “Without this integration of the systems investigating a sample with scanned probe microscopy required removing the sample from the micro-Raman spectrometer. This meant that the exact region that was being interrogated by Raman could not be effectively correlated with the chosen SPM imaging technique.”

“Another aspect of optical integration is that SPMs can measure forces but they cannot measure distribution of light in micro-lasers silicon-based wave guides fluorescently stained biological materials etc.” explains Lewis. “For example there are many important advances occurring in the application of photonics to silicon structures and plasmonic metals. In the past these photonic structures were in the micrometer range now they are nanometric.”

The Nanonics platform can be used for structural and photonic characterization as well as the structural and chemical characterization that is available with AFM and Raman integration.

For these applications Nanonics Imaging is the innovator of AFM and NSOM systems including dual tip/sample scanning AFM systems the industry’s first NSOM-AFM cryogenic systems integrated Raman-AFM systems multiprobe AFM and SEM-AFM systems. The company also holds patents to the largest range of unique nano-probes. These probes form a NanoToolKit for its unique characterization platforms with a variety of tasks such as for nanophotonics plasmonics nanochemical imaging and even nanochemical deposition based on its singular NanoFountainPen technology. The company is focused on full integration of AFM technology with optics chemical imaging and other analytical tools.

The Nanonics MultiView AFM-NSOM microscope with its free optical axis on a standard micro-Raman now makes it possible to truly integrate the separate worlds of Raman and AFM/NSOM nanocharacterization which has led to a new era in high-resolution Raman spectroscopy. Facilitating this integration is not only the geometry of the AFM/NSOM platform but also a new generation of AFM glass probes that have very unique characteristics – such as hollow glass probes with cantilevered nano-pipettes for material deposition probes with glass surrounding a single nanowire in the middle for ultrasensitive electrical measurements or dual wire glass probes for thermal conductivity and thermocouple measurements. Glass probes are ideal for Raman integration because of their transparency to laser light and no Raman background. They also expand outward allowing unprecedented correlation of Raman and AFM also permitting multiple probes to be brought easily together which is very difficult with a standard AFM

## Negative-Stiffness Vibration Isolation – Enabling AFM and Micro-Raman to Function as an Integrated Platform

Underlying this pioneering integration AFM with micro-Raman is negative-stiffness vibration isolation developed by Minus K Technology Inc. What negative-stiffness isolators provide is really quite unique to SPM-Raman and other NSOM systems. In particular improved transmissibility of a negative-stiffness isolator – that is the vibrations that transmit through the isolator relative to the input floor vibrations. Transmissibility with negative-stiffness is substantially improved over air systems and over active isolation systems. Negative-stiffness isolators employ a unique - and completely mechanical - concept in low-frequency vibration isolation. Vertical-motion isolation is provided by a stiff spring that supports a weight load combined with a negative-stiffness mechanism. The net vertical stiffness is made very low without affecting the static load-supporting capability of the spring. Beam-columns connected in series with the vertical-motion isolator provide horizontal-motion isolation. The horizontal stiffness of the beam-columns is reduced by the "beam-column" effect (A beam-column behaves as a spring combined with a negative-stiffness mechanism). The result is a compact passive isolator capable of very low vertical and horizontal natural frequencies and very high internal structural frequencies. The isolators (adjusted to 1/2 Hz) achieve 93% isolation efficiency at 2 Hz; 99% at 5 Hz; and 99.7% at 10 Hz.

“Before negative-stiffness vibration isolation was employed AFM used in conjunction with micro-Raman systems could not maintain adequate imaging integrity while measuring at the nanoscale level” said Lewis. “Vibration isolation is absolutely necessary for the system’s successful performance and negative-stiffness isolation has enabled AFM and micro-Raman to function as a truly integrated platform.”

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