Improving nanoimaging of µ-Raman/AFM systems

Negative-stiffness vibration isolators can easily support the heavy weight of a combined AFM/micro-Raman system, and isolate it

BY JIM McMAHON
Zebra Communications
Simi Valley, CA
www.zebracom.net

The need for precise vibration isolation with scanning probe microscopy (SPM) and nearfield 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 displacements of a very few angstroms or nanometers, an absolutely stable surface must be established for the instrument. Any vibration coupled into the instrument's mechanical structure will cause vertical and/or horizontal noise and thus reduce the system's ability to measure high resolution features. And while the vertical axis is the most sensitive for SPMs, they can also be quite sensitive in the horizontal axis.

Traditional isolation

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. Benchtop air systems provide limited vertical and horizontal isolation.

Also at a disadvantage are systems based on active isolation, known as electronic force cancellation. Such systems use electronics to sense the motion and then add 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.

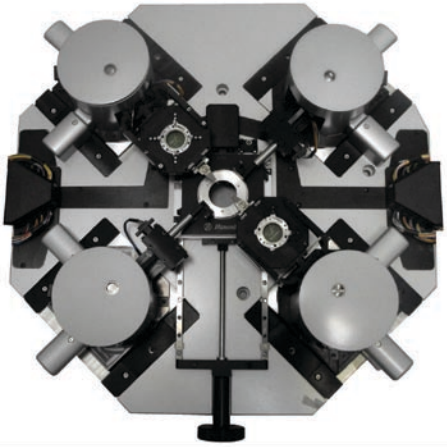

Fig. 1. The MicroView4000 platform from Nanonics Imaging is the basis for AFM-Raman integration.

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

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 even a very flat sample is scanned, it is hard to keep the lens-to-sample distance 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 artifactual intensity variations in the Raman unrelated to the chemical composition of the sample.

This is even more pronounced with rough samples, and standard methods of auto-focus are simply not accurate enough for a host of problems 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 AFM, 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 nanoscale. 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.

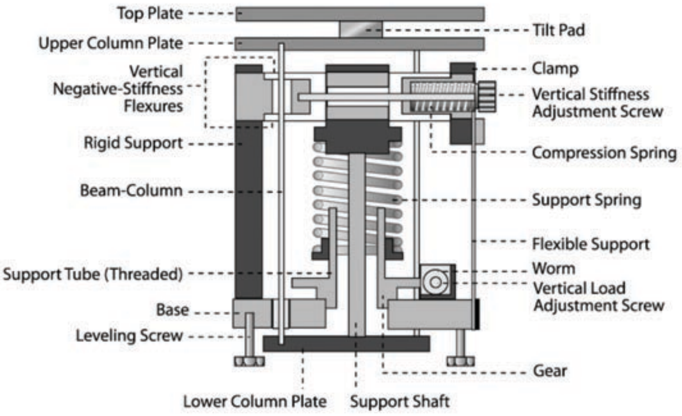

Fig. 2. The negative-stiffness isolator concept developed by Minus K Technology makes it possible for a heavy AFM-Raman system to be immune to shocks that would make nanoprecision impossible.

The AFM consists of a microscale 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 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 (see *Fig. 1*).

This advantage addresses critical problems in Raman including resolution and intensity comparisons in Raman images while permitting online 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.

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 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 nanopippets 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.

NSVI enables integration

Underlying this pioneering integration AFM with micro-Raman is negative-stiffness vibration isolation, developed my Minus K Technology. 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, completely mechanical concept for low-frequency vibration isolation. Vertical-motion isolation is provided by a stiff spring that supports a weight load, combined with a negative-stiffness mechanism (see *Fig. 2*).