Journal of
Scanning Probe Microscopy
Vol. 1, 51–54, 2006

NanoLaboratory Concept: A Platform Combining Advanced Scanning Probe Microscopy and Non-Scanning Probe Microscopy Methods

Denis Andreyuk

NT-MDT Co., Building 2, room 431, Technopark, South Industrial Zone, passage 4922, Zelenograd, Moscow, Russia

The NanoLaboratory concept is described as the technical platform for joining of advanced scanning probe microscopy to the most modern non-scanning probe microscopy methods. Specific examples of how current limitations can be overcome in scanning probe microscopy itself (improvement of system stability for nanomanupulations and nanolithography) and of new possibilities gained from a multidisciplinary approach (scanning probe microscopy-based tomography and ultra-high resolution optical methods) are considered in terms of a scanning probe microscopy development perspectives.

Keywords: Scanning Probe Microscopy, Nanolaboratory, Drifts Compensation in SPM, SPM-Based Tomography, Tip-Enhanced Raman Scattering.

Market requirements are the major power driving development of any scientific instrumentation. From this point of view scanning probe microscopy (SPM) now seems to be between Scylla and Charybdis. On one hand, traditional nanotechnology applications have more and more advanced requirements, forcing manufacturers to develop highly specialized systems. On the other hand, the number of scientific disciplines that are interested in nanoscale research is growing in an avalanche-like manner. Thus the flexibility of SPM-based systems and compatibility to other methods become a bottle-neck, limiting the widespread implementation of SPM into all branches of scientific research.

Here the NanoLaboratory platform is presented (NTEGRA platform). It allows creating of advanced SPM systems fitting the highest requirements of some specialized applications, in the meantime keeping enough flexibility to be combined to the non-SPM methods. The term "platform" here means that all NTEGRA line instruments share some common parts in the SPM realization. Moreover, they all are driven by the same electronics module, and there is one universal software package, so that all applications share the same interface logic. An important point is also the fact that all instruments are created by the same team of engineers and programmers, thus sharing the same "platform philosophy".

To prove the advantages of such a philosophy, some technical applications will be considered in terms of limitations that can be either totally or partially overcome within the NanoLaboratory platform.

The first examples are nanolithography and nanomanipulations. These applications are particularly demanding to the SPM itself, since the goal is not only to visualize the small area (tens of nanometers in the most critical cases). There should be a possibility to rescan the same area (thus repeatability must be high enough), and, most importantly, there should exist the possibility to perform local influence (such as mechanical or electrical) with extremely high accuracy. In other words precise positioning of the probe relative to the sample at every moment is the most significant SPM value from the nanolithography and nanomanipulations points of view.

The most serious problem here is that any SPM system has some intrinsic drift, causing unwanted (and uncontrolled) displacement of the probe relative to the sample. The SPM quality can be expressed as how low the uncontrolled system drifts are. There are two most prominent causes of probe-to-sample displacement: piezoceramic imperfections and thermodrift. In the NanoLaboratory, both SPM effects have been substantially compensated.

The common way to compensate scanning elements imperfections (creep, hysteresis, and so on) is to integrate closed-loop sensors, tracking real piezodriver displacement. (Along with a closed-loop correction, a software correction is often used. It is also realized within the NTEGRA platform.) This is a good solution, and most SPM manufacturers offer it. But any closed-loop sensor

COMMUNICATION

always generates its own noise, affecting the measurements quality. On submicrometer scanning fields, it becomes crucial. Thus the size of the smallest area that can be scanned with the closed-loop control switched on is the criterion of the sensor and integration quality. On most commercially available systems, the closed-loop sensors should be switched off below 300–500 nm. NTEGRA line SPMs provide confident closed-loop control down to 50 nm. In addition, in most NTEGRA configurations, a low-noise sensor for the Z-direction is available too, since precise control of vertical probe movement is absolutely essential for many nanomanipulation experiments.

Currently there are no common approaches to overcome thermodrift, i.e., drift caused by unequal thermal expansion/compression of the SPM parts. They actually represent a serious problem. From quantitative evaluation made in Ref. [1], it becomes obvious that 50 nm per Kelvin of temperature difference is a reasonable value. In working SPM device, there always exist some local source of heat (e.g., integrated electronics components, lasers, etc). Moreover, even under climate-controlled conditions, room temperature differences of 2–5 K are usual due to slow airflow. As a result, in the best commercially available SPMs typical drift when working at room temperature is 10–15 nm/h. The problem is even more prominent when the experiment requires heating or cooling of the sample. In that case displacement usually is not less than 30–50 nm/K. In other words heating the sample from room temperature to 120 °C normally causes 3–5 μm displacement!

There are several solutions developed within the NTEGRA platform to decrease the SPM thermodrift. One is the so-called self-compensation principle. The main issue here is thoroughly calculated system geometry and material combination (thermal expansion and thermal conductivity coefficients are mainly taken into account). As a result the overall probe-to-sample displacement is minimized, because expansion of any single part is always compensated by expansion of some other part of the system. Another solution is the measurement chamber's design, providing almost no temperature gradient between the probe and the sample. For high-temperature experiments the important feature is that the scanner is spatially isolated from the measurement chamber, thus it is not heated directly. The SPM registration module design has been changed to reduce the laser noise, and sophisticated whole-system cooling has been developed. The result can be expressed quantitatively: the room temperature drift in the NTEGRA Therma system is 3–5 nm/h (Fig. 1). When working at a temperature other than the room temperature, the drift is as low as 10–15 nm/K. It is a safe bet to say that the system stability is much better than that achievable in any other commercial system to date, and this particular characteristic makes it the system of choice for nanolithography and nanomanipulation applications.

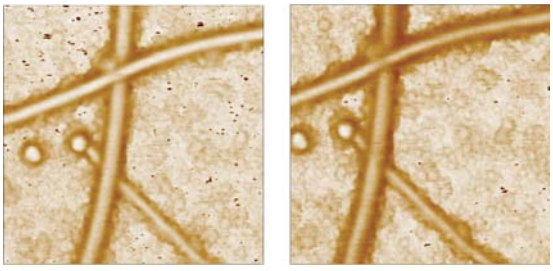

Fig. 1. Nanoparticles coupled to carbon nanotubes. The same area (200×200 nm) was imaged repetitively for 7 h. Overall XY displacement of the marker feature (single nanoparticle) for 7 h was as small as about 35 nm. Images obtained by Dr. A. Temiryasev, NT-MDT. Sample courtesy of Dr. H. B. Chan, Department of Physics, University of Florida, USA.

Being the system developed for applications, where the long-term stability is required and experiments at changing temperature are of high importance, NTEGRA Therma also represents an example of a highly specialized system within the NanoLaboratory platform. The next step is to consider the functionality appearing when SPM is combined with non-SPM approaches. It is worthwhile to focus on cases where the functionality of a combined system is greater than the sum of functionality of the two instruments working separately, i.e., when the combination opens up some new perspectives.

It is well-known that most of SPM methods study the surface of the sample. SPM tomography allows the expansion of the power of SPM multiparametric experiments into the sample volume. The principal scheme of the SPM tomography setup is shown in Figure 2. Two instruments are working successively: ultramicrotome and SPM. Ultramicrotome's diamond knife removes a slice from the sample, opening the surface for SPM imaging or measurement. Then the next slice is removed and the next cycle begins. A series of SPM images can then be reconstructed into a 3D model, where spatial distribution of parameters of interest can be visualized and studied quantitatively. The thickness of each section can be as small as 20 nm and this is a resolution limit for the Z (third)-dimension.

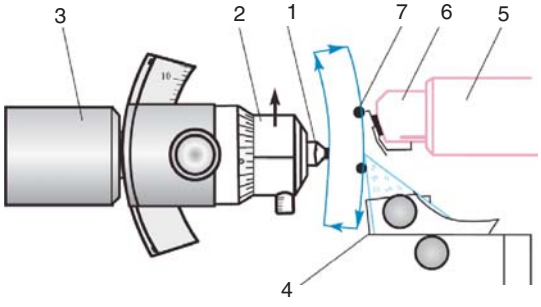

Fig. 2. Principal scheme of the AFM tomography setup: 1, sample; 2, sample holder; 3, movable ultramicrotome arm; 4, ultramicrotome knife; 5, AFM scanner; 6, probe holder; 7, AFM probe.

COMMUNICATION

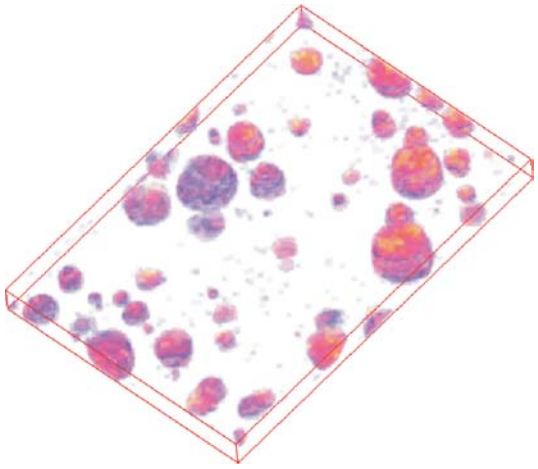

Fig. 3. 3D model of ABS/PA6 (acrylonitrile–butadienestyrene/polyamide 6) polymer blend structure (8.0 × 5.6 × 0.6 um, sectioning step was 40 nm). SPM images obtained in phase detection microscopy mode. Images obtained by Dr. A.Efimov, NT-MDT. Sample courtesy of Institut f. Polymere, ETH Zurich, Switzerland.

Currently there are two branches of science that are urgently interested in SPM tomography. First is the creation of new materials, especially nanocomposite polymers. Figure 3 shows a 3D model of a polymer blend as reconstructed after phase detection microscopy with a sectioning step of 40 nm. Distribution of silica or carbon nanoparticles within the polymer matrix can be visualized this way. From the material testing point of view, a large variety of SPM techniques offer additional possibilities, since electrical and magnetic properties of the material sample can be reconstructed as well.

Another field where SPM tomography can be very useful is in the Life Sciences. Some reports have already been published concerning applicability of SPM to epoxy resin embedded biological samples.[2] There is no doubt that SPM tomography is a very promising tool for such areas as developmental biology, neuroscience, and cancer medicine—for all fields where 3D visualization is of most importance.

Optical properties can be studied by conventional optical microscopy-based methods. The main limitation common for all such approaches is the diffraction of light, which affects the spatial resolution. A confocal scheme and some advanced techniques can greatly improve signal-to-noise ratio (e.g., such techniques for fluorescence imaging as multiphoton excitation or total internal reflection), but the in-plane (*XY*) resolution cannot be better than approximately half of the light wavelength, i.e., about 200 nm, in the best confocal systems. The same is the limit of theoretically achievable resolution for microscopy-based local spectroscopy techniques (e.g., Raman microspectroscopy). In fact, the *XY* resolution in commercial micro-Raman systems currently is about 300–500 nm.

It should be mentioned that conventional microscopy-based micro-Raman systems have relatively high sensitivity thresholds. If only a few molecules of interest are present within the beam spot, they usually will not provide a Raman signal strong enough to be detected (remember that about 1 of 10^7 incident photons is scattered by Raman effect).

Scanning near-field optical microscopy (SNOM) employing the unusual properties of light passed through the subwave aperture overcomes the diffraction limit. In that case a resolution of 30–50 nm can be achieved. Light intensity is the main factor limiting SNOM applicability. After light has gone through the aperture, its intensity decreases by factor of 10^4–10^5. This is why Raman spectroscopy can not be performed via SNOM. For the same reason, even weak fluorescence registration (e.g., single molecules detection) is substantially obstructed within this method.

The most promising approach to increasing the signal intensity is to use effects of local electromagnetic field enhancement. The phenomenon is that in close proximity of nanometer scale asperities, the light properties significantly change, greatly enhancing Raman scattering and some other optical effects. For silver or gold asperities the theory predicts local Raman scattering enhancement by a factor of 10^{12}.[3]

The so-called SERS technique (surface-enhanced Raman scattering) nowadays is widely used for single molecule detection by conventional Raman microscopy. The idea is that if molecules of interest are homogenously distributed on the surface of a special substrate (usually there are fixed Ag nanoparticles), some molecules would sit near the SERS-active particles. When such molecules are illuminated, they provide a detectable signal. Two

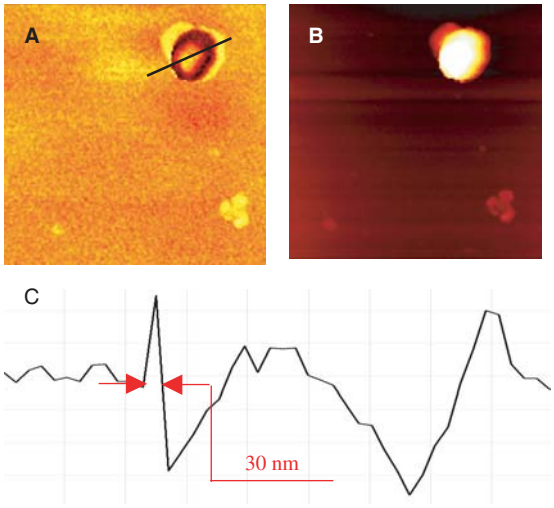

Fig. 4. Latex particles imaged synchronously by confocal laser microscopy (A) and atomic force microscopy (B). Apertureless near-field effects occurring between the super sharp DLC tip and the sample provide in-plane optical resolution almost as good as that of AFM (about 30 nm as marked by arrows on the line profile, C). The scan size for A and B is 2.8 × 2.8 μm. Images obtained by Dr. K.Mochalov, NT-MDT.

principal limitations are evident in this method. First, only very small particles or very thin films can be the objects for such observations, and they must be deposited on the SERS-active substrate. Second, single molecules can be detected this way, but they cannot be identified. In other words, the Raman signal (even enhanced) can be mapped only with diffraction-limited spatial resolution. For example, two molecules occurring near the same SERS-active nanoparticle 40 nm apart can both be detected but can never be resolved.

Alternative technique exploiting local field enhancement effects is TERS (tip-enhanced Raman scattering). Metal (silver or gold) nanoscale asperities are associated with the SPM probe. When positioned within the beam, the TERS-active probe generates a strong Raman signal only from the small volume around the tip. As the enhanced Raman signal is many orders of magnitude more intensive than the signal from other illuminated areas, spatial resolution of the method is confined to that area where enhancement occurs. Thus, TERS is an appropriate technique both for detecting and identifying single molecules by their spectral properties. On single-walled carbon nanotubes, Raman signal mapping with 40-nm *XY* resolution has been recently demonstrated by using a NTEGRA Spectra system.[4]

An important issue here is accurate coordination of optical and SPM parts of the system. Figure 4 demonstrates the optical image of a latex particle with in-plane resolution of 30 nm gained simultaneously with an AFM image. Extremely high optical resolution (appearing due to the apertureless near-field effects on the super sharp DLC tip) would not be possible if scanning and detection signals had not been fully synchronized.

Summarizing all facts, the most significant NanoLaboratory concept's advantages can be concluded as two principles. (1) Highly specialized systems fitting the most critical requirements can be created on the NanoLaboratory platform. (2) The platform is, nevertheless, oriented to the in-depth integration of SPM and non-SPM methods, which in some cases has resulted in a qualitative breakthrough.

References and Notes

1. B. Mokaberi and A. A. G. Requicha. *Proceedings of the IEEE Int'l Conf. on Robotics and Automation*, New Orleans, LA **(2004)**.
2. N. Matsko and M. Mueller, *J. Struct. Biol.* 146, 334 **(2004)**.
3. A. Hartschuh, M. R. Beversluis, A. Bouhelier, and L. Novotny Philos, *Trans. A Math. Phys. Eng. Sci.* 362, 807 **(2004)**.
4. http://www.ntmdt.com/download/catalog_NTEGRA.pdf.

Received: 28 January 2006. Revised/Accepted: 31 March 2006.