NenoVision

Application Note

In situ SEM/AFM characterisation of hybrid structures made of graphene-veiled gold nanoparticles for bio sensing

The combination of gold nanoparticles and graphene veiling is a novel approach to fabrication of active substrates suitable for Surface Enhanced Raman Spectroscopy (SERS). Graphene can serve as a pin-hole passivation layer, which prevents plasmonic nanostructures from oxidizing.

The level of the contact between Au particles and graphene plays a significant role in the sensitivity of SERS [1]. Therefore, it is important to know the way a graphene membrane veils a single nanoparticle or a cluster of such nanoparticles. The distribution of the nanoparticles under the graphene membrane as well as the surface topography can be easily determined using CPEM – Correlative Probe and Electron Microscopy™.

CPEM, which combines both SPM and SEM techniques, is a revolutionary characterization method for nanoworld imaging. This novel approach makes it possible to obtain high-resolution images (SPM and SEM) of the same area at the same time and in the same coordination system.

While LiteScope™ SPM (AFM) can image the surface of a graphene layer on top of nanoparticles, SEM can image nanoparticles under the graphene layer, this layer being close to transparent to an electron beam.

Fig. 1 provides a comparison of these two images during which instant information about the shape of the graphene membrane over the specific nanoparticle was obtained. CPEM enables to clearly determine the number of particles in clusters and distinguish if the graphene membrane is only supported by particles or if it is wrapped around the particles (Fig. 2).

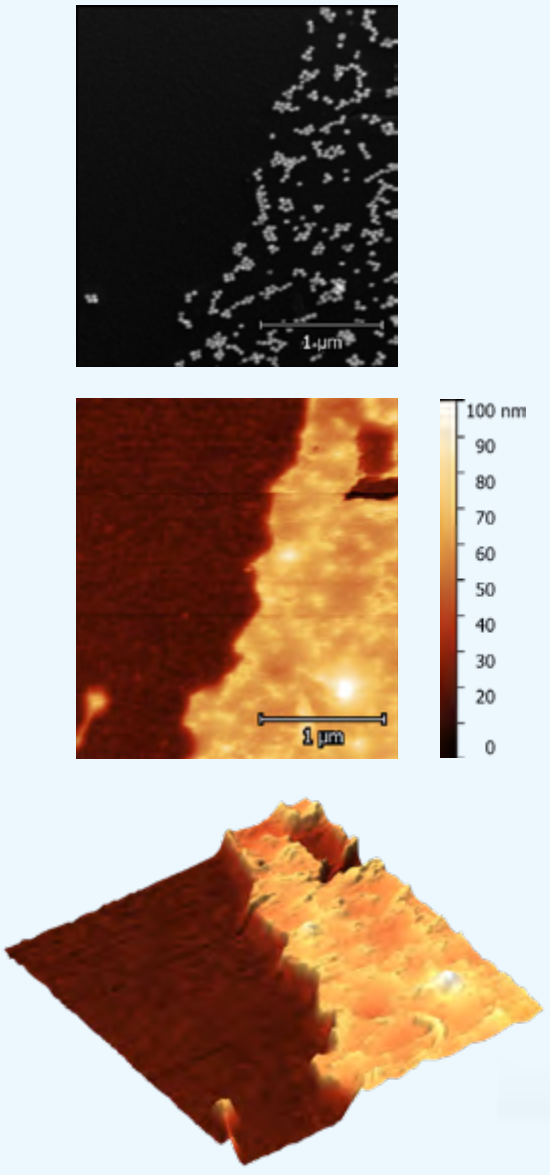


Figure 1: SEM and SPM (AFM) images complemented by a 3D visualization of a graphene membrane on top of Au nanoparticles. SEM reveals the exact position of the particles while AFM shows how graphene covers single particles or their clusters.

Experiment

Gold nanoparticles (ø 40 nm) from a colloidal solution were deposited on a silicon surface with a 285 nm thick layer of SiO_2. Subsequently, the array of gold nanoparticles was overlaid by graphene produced by Chemical Vapor Deposition (CVD) [2]. The sample was measured in an FE SEM (HV 15 kV, SE detector) with an integrated AFM - Litescope™. Surface investigation was carried out in the AFM tapping mode using an Akiyama probe.

Summary

AFM images show that graphene does not completely wrap the nanoparticles. This results in reduced SERS effect.
It was successfully shown that CPEM is a unique method for studying surface structures.
In addition, as CPEM uses an innovative combination of different SPM modes of LiteScope™ and various SEM detectors, it provides more than one type of information.
It opens a completely new field of correlative imaging as well as many special applications.
Moreover, direct scanning of the surface area displayed by the SEM makes navigation and finding specific locations and structures simple.

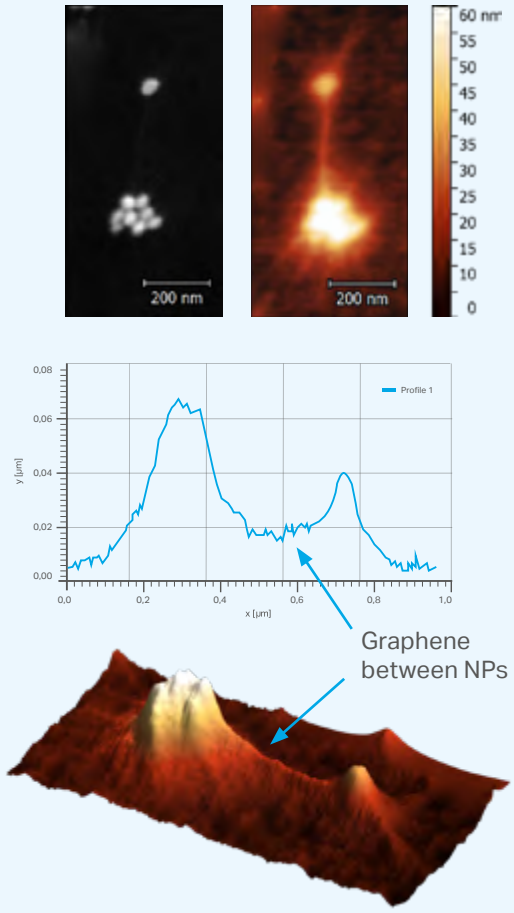
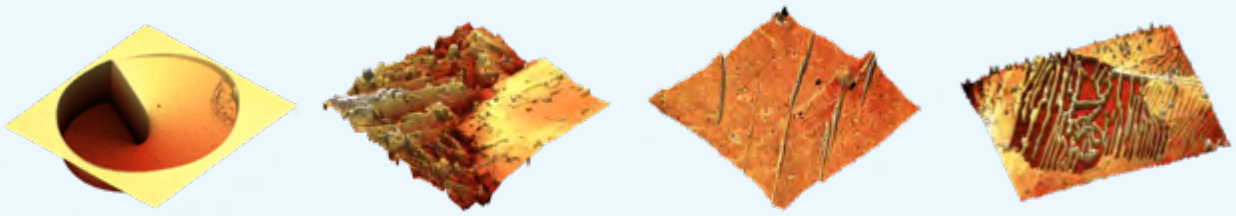


Figure 2: SEM and AFM images of a graphene "curtain" between a single nanoparticle and a cluster. Images were measured by a CPEM – at the same time and in the same coordination system.

Image Gallery



FIB milled vortex structure

Wet etched Al surface

Fine-grained Cr steel

White cast iron (etched)

[1] Xu, W., Xiao, J., Chen, Y., Chen, Y., Ling, X., & Zhang, J. (2013). Graphene-veiled gold substrate for surface-enhanced raman spectroscopy. Advanced Materials, 25(6), 928–933.

[2] Prochazka, P., Mach, J., Bischoff, D., Liskova, Z., Dvorak, P., Vanatka, M., … Sikola, T. (2014). Ultrasmooth metallic foils for growth of high quality graphene by chemical vapor deposition. Nanotechnology, 25(18), 185601.

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