Seeing the invisible - ultrasonic force microscopy for true subsurface elastic imaging of semiconductor nanostructures with nanoscale resolution.

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ABSTRACT

In this paper we produce first unambiguous SPM images that utilise ultrasonic force microscopy (UFM) approach for imaging of internal morphology of two dissimilar high stiffness solid state nanostructures - 50 nm thick graphite flakes on the patterned substrate and iii-v InAs/GaAs semiconductor quantum dot structures under atomically flat GaAs capping layer. Moreover, by analysing the imaging process, we show that the imaging mechanism in reported so far subsurface imaging methods is indeed the elastic field produced by the indention of dynamically stiffened cantilever-tip system due to high vibration frequency, with detection due to the nonlinear tip-surface interaction.

Keywords: atomic force microscopy, ultrasonic force microscopy, subsurface imaging, graphene, nanostructures

1 INTRODUCTION

Scanning probe microscopes (SPM's) play indispensable role in modern nanoscale material science by enabling imaging of surfaces with close to atomic resolution. Unfortunately, ability of SPM's to look below the surface is unavoidably limited. A successful attempt of combining SPM with ultrasonic imaging capable of viewing inner structure of materials and devices resulted in Ultrasonic Force Microscope (UFM) [1, 2] and its further modifications including Heterodyne Force Microscopy (HFM) [2, 3] and HFM-like methods [4] that were shown to have some subsurface capability for voids as well as for structure of soft material like polymers [2-5].

At the same time the true subsurface imaging in solid state nanostructures composed of stiff materials has yet to be reliably demonstrated. Moreover, some misconceptions still exist as to how the wave propagation of ultrasonic waves contributes to the imaging, e.g. it was suggested [4] that multiple interfering waves are generated in the sub micrometere size studied volumes near the SPM tip, that can not feasible given millimetre length scale of ultrasonic wavelength used that is several orders of magnitude larger than volumes involved.

2 NANOMECHANICAL MAPPING IN SPM VIA ULTRASONIC VIBRATIONS

In ultrasonic assisted SPM (UFM, HFM or related methods) [2-6] a sample is vibrated at very high frequency $f_{UFM} >> f_c$ (typically between 2 - 60 MHz) and amplitude modulated at low (few kHz) frequency. Due to the high dynamic rigidity of the AFM tip-cantilever system, a nanoscale tip cannot move with the sample vibration, but instead elastically deform the sample at high frequency. If one assumes the concentrated mass and stiffness for the cantilever, its dynamic stiffness increases as $(f_{UFM}/f_c)^2$, whereas taking into account that cantilever mass and spring are distributed, dynamic stiffness of the cantilever can be approximated as $k_{dyn}=k_c\ (f_{UFM}/f_c)^{3/2}$. Substituting values for cantilever and UFM frequency and used in this study, we obtain $k_{dyn}\approx 1100$ N/m, expanding range of stiffness accessible by FMM by three orders of magnitude [3].

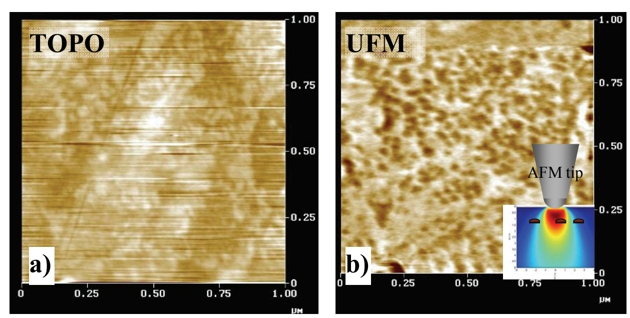

Figure 1: a) AFM topography and b) UFM elasticity image of InAs QD's on GaAs substrate partially caped with 2 nm GaAs layer. UFM images reveal features similar to topography but provide better discrimination of QD structures (eg. in fuzzy areas in topography indicated by arrows). InAs has lower elastic stiffness moduli than GaAs and seeing as darker in UFM images. Image width 1 µm. inset – map of Hertzian stress field propagating inside the sample.

The oscillating contact force is subsequently “rectified” owing to the extreme nonlinear force-vs-distance dependence of a tip-surface contact resulting in a net force at kHz modulation frequency that is easily detectable by the AFM cantilever. In HFM [4, 5] the nonlinearity produces the “beating” frequency much lower than the excitation frequency. UFM and HFM was shown to have an excellent material contrast to nanoscale surface features of semiconductor nanostructures ranging from quantum dots and superlattices to engineering ceramics and composites and an example. A good example of this is given in figure 1 where InAs quantum dots on the GaAs substrate and partly overcoated by thin layer of GaAs are shown. UFM provides excellent material contrast not directly related to the topography.

Another very useful feature of UFM is that it eliminates sample-tip friction as the solid-solid contact between SPM tip and the sample is broken for the part of oscillation period, thus allowing gentle imaging of the sample similar to the tapping mode.

3 NANOMECHANICAL MAPPING IN SPM VIA ULTRASONIC VIBRATIONS

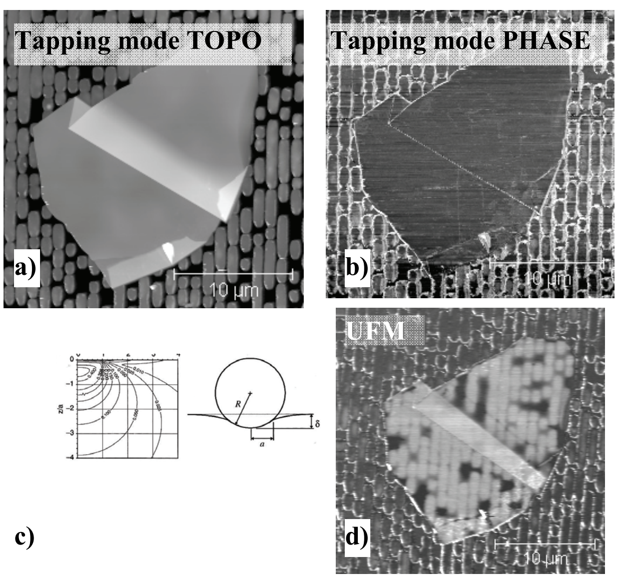

Figure 2: a) AFM tapping mode topography and b) AFM tapping mode phase image of 50 nm thick graphite “slab” on patterned COC (cyclic olefine). No subsurface contrast is visible in these images. d) same graphite flake imaged using UFM (elastic stiffness) mode - areas in contact with COC pillars are clearly visible via stiff 50 nm slab of graphite. c) graphs of calculated elastic stress propagating from the tip in contact with the graphite – same graph is applicable to the elastic field propagating from the slab support

In Figure 2 shown first true subsurface images of thick graphene flake (50 nm) on the pattenered polymeric substrate. While standard AFM images)tapping mode amplitide and phase) fail to observe any variations on the flake, UFM easily looks through revealing details of the subsurface contact between the flake and the substrate.

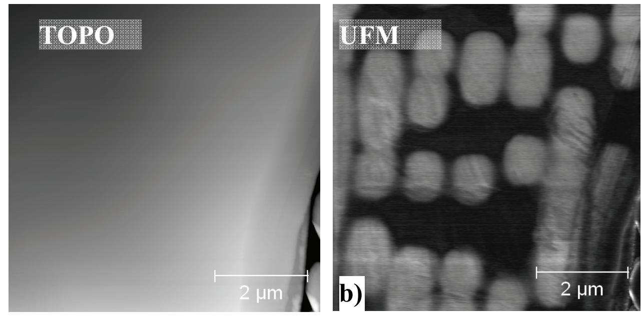

Figure 3: a) AFM topography and b) UFM stiffness image of the graphene flake at figuere 2 at a higher magnification.

Figure 3 shows higher resolution of the same flake where fine details of the contact can be observed. Lateral resolution to the UFM elastic field is on the order of 50 nm that is consistent with the thickness of the graphite slab. Ultrasonic frequency was 4 MHz corresponding to the in plane acoustic wavelength in graphite of 4 mm, suggesting that all parts of graphite within this few this few µm region are vibrating in phase.

4 ULTIMATE RESOLUTION AND PROBED DEPTH IN UFM AND HFM SUBSURFACE IMAGING

The wavelength of ultrasound in all used so far attempts for subsurface imaging ranged from sub MHz to few MHz. In most semiconductor materials with speed of sound on the order of 5,000 m/s that corresponds to the mm wavelength, and even in polymers (with speed of sound of 1.5-2,000 m/s) that is at best 100 µm. Imaging of structures with the dimensions of tens of nm at the depths of 100 nm makes it clear the near-field nature of ultrasonic (UFM, HFM, etc) SPM subsurface imaging.

Therefore the highest resolution of subsurface UFM and HFM will be for the samples with nanoscale near-surface features. Such sample is presented in figure 4 where iii-v InAs/GaAs semiconductor quantum dot structures under atomically flat GaAs capping layer.

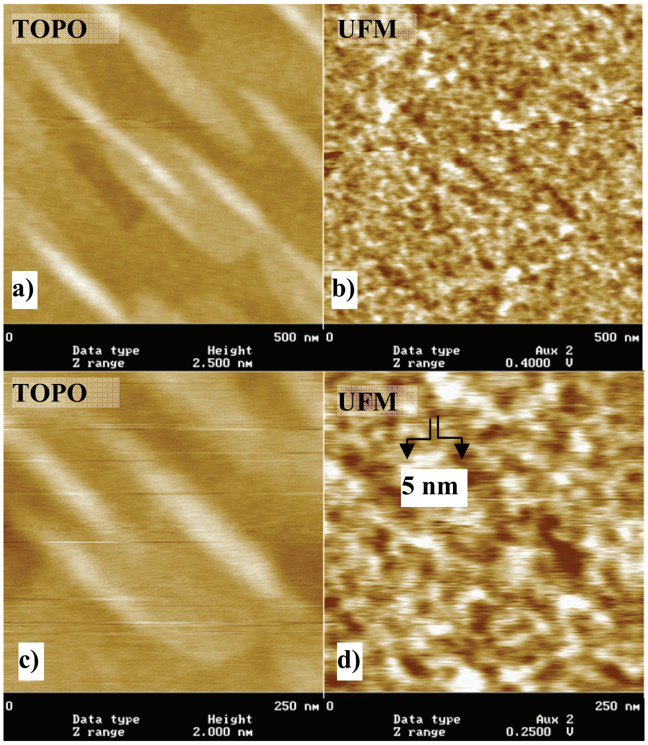

Figure 4: A) AFM topography and d) UFM elasticity of InAs QD's fully capped by 50 A GaAs layer. Both images are obtained simultaneously, and while AFM shows only atomically flat terraces, UFM clearly reveals QD's under the capping layer (image width 500 nm). e, f) corresponding magnification of area shown in c) (image width 250 nm). Smallest feature in UFM subsurface image of InAs QD's is ~ 5nm.

The total absence of surface topographical contarst on the GaAs terasses and simultaneous excellent contrast to subsurface InAs quantum dots unambiguously demonstrate to our knoeledge for the first time the 5 nm resolution to these "soft" elastic inhomogeneities (as opposed to cracks, voids or delaminations.

Moreover, by analysing the imaging process, one can show that the imaging mechanism in reported so far subsurface imaging methods [4-6] is indeed the elastic field produced by the indention of dynamically stiffened cantilever-tip system due to high vibration frequency, with detection due to the nonlinear tip-surface interaction.

The subsurface sensitivity is identical, whether UFM mode (detection of ultrasonic vibration) or HFM mode (mixing of two vibrations) is used, as long as studied volume (100 - 2000 nm) is much smaller than wavelength of ultrasound used (500 - 2000 μm at 2-10 MHz). Phase information available in HFM could play some role in the viscoelastic relaxation imaging [4], but for subsurface mapping it produces a minute correction due to a very large scale difference of ultrasonic wavelength (~mm) and the imaged volumes (~nm). The ultrasonic lubricity (vanishing of friction in nonlinear regime) is a great bonus of UFM and HFM derived methods minimising their damage to the surface similar to the tapping SPM mode.

5 CONCLUSIONS

In conclusion, a unique feature of applying UFM methods for subsurface imaging of solid state and semiconductor nanostructures is that it demonstrates enables nanoscale imaging with down to 5 nm resolution by visualising elastic properties subsurface nanostructures in their natural non-disturbed environment. In particular, capped QD's we directly observed in UFM were smaller than surface QD's that confirms the observation obtained via destructive TEM investigation. Further expansion of this methodology, challenges and potential applications are also discussed.

REFERENCES

[1] Kolosov, O. V.; Yamanaka, K. Japanese Journal of Applied Physics Part 2-Letters 1993, 32, (8A), L1095-L1098.

[2] Yamanaka, K., H. Ogiso and O. Kolosov, UFM for nanometer resolution subsurface imaging, APL 64(2): 178-80, (1994);

[3] A. Briggs and O. Kolosov, Acoustic Microscopy, 2nd edition, Oxford University Press, 2010.

[4] M T Cuberes, H E Assender, G A D Briggs and O V Kolosov, Heterodyne force microscopy, J. Phys. D: Appl. Phys. 33 (2000) 2347.

[5] Diebold, A. C., Subsurface Imaging with Scanning Ultrasound Holography, Science 310(5745): 61-62, (2005).

[6] Tetard L; Passian A; Thundat T. Nat Nano 5, (2), 105-109.