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Near-Field Acoustic Holography as a High Resolution Sub-Surface Imaging System on Scanning Probe Microscopy Platform: *Seeing the Invisible!*

Gajendra Shekhawat* and Vinayak P. Dravid#*

*Institute for Nanotechnology, 1161 Cook Hall, Northwestern University, Evanston, IL 60208
Material Science and Engineering, 1141 Cook Hall, Northwestern University, Evanston, IL

We have developed a novel near-field acoustic holography (NFAH) technique which combines the nanometer-scale spatial resolution of conventional scanning probe microscope (SPM) with the surface and subsurface imaging capabilities. This technique fills the critical void in characterization and investigation of the static and dynamic mechanics of nanoscale systems and address emerging issues in imaging and analysis of diverse "embedded" nano and microscale structures, and engineered systems. The NFAH development integrates three major approaches: a unique combination of scanning probe microscope platform (which enjoys excellent lateral and vertical resolution) coupled to microscale ultrasound source and detection (which facilitates "looking" deeper into structures, section-by-section) and a novel holography approach (to enhance phase resolution and phase coupling in imaging).

Existing far-field ultrasonic or acoustic microscopy techniques [1] suffer from spatial resolution limitations. Moreover, all far field microscopies lack: resolution Limitations due to Rayleigh limit: $0.51\lambda/N.A.$, coupling fluid attenuation $\sim f^2$, and impedance mismatches. Recently ultrasonic force microscopy [2-5] has been widely used to map the elastic properties of soft and hard surfaces. It also provides quantitative analysis of surface mechanical properties. But, it lacks the sub-surface imaging and sub-surface defect identification such as embedded voids, cracks, and phase distribution, among others.

In NFAH approach, a high frequency (~ 100's of KHz to several MHz) acoustic wave is launched from the bottom of the specimen, while another wave is launched on the AFM cantilever, albeit at a slightly different frequency. The interference of these two waves would nominally form so-called "beats" and "product frequencies" which is monitored by the AFM tip, which itself acts as an antenna for both phase and amplitude of the beats and product frequencies. As the specimen acoustic wave gets perturbed by sub-surface (and surface) features, especially its phase, the local acoustic interference is very effectively monitored by the AFM tip. Thus, within the near-field regime (which enjoys superb lateral and vertical resolution), the acoustic wave (which is non-destructive and sensitive to mechanical/elastic variation in its "path") is fully analyzed, point-by-point, by the AFM acoustic antenna in terms of phase and amplitude. Thus, as the specimen is scanned across, a pictorial representation of acoustic wave's perturbation is fully recorded and displayed, to offer "quantitative" account of internal microstructure of the specimen. The system is operated in near-contact mode to acquire the high resolution sub-surface images.

The efficacy of NFAH to achieve high-resolution sub-surface imaging is presented in Fig. 1. The NFAH phase image clearly reveals the embedded gold nanoparticles underneath the polymer composite structures. The model sample is a polymer/metal composite system based on the adsorption of colloidal gold particles from a dilute aqueous suspension to the surface of poly (2-vinylpyridine) (PVP). The particles have an average diameter of 15 nm and are well dispersed on the film surface. Top layer is another polymer film about 500 nm thick. In contrast to the (expected) featureless AFM topographic image, Figure 1b shows the spectacular example of high resolution sub-surface imaging of the sub-surface nanoparticles. The gold particle size varies from 15-20 nm across the image. Dark contrast in the NFAH image is due to time dependent phase delay of the acoustic waves reaching at the sample surface, which shows the change in the viscoelastic response. Figure 2 shows another potential application of NFAH approach in direct imaging of buried magnetic nanoparticles in silica shells. Topography scan (fig. 2a) shows uniform distribution of silica particles spread over silicon dioxide surface with size varying from 50-100 nm. Cobalt magnetic particles from 15-25 nm are enclosed in silica core shell.

Extending the application of NFAH in studying the sub-surface defects in biomaterials, we choose red blood cells (RBC) and infected them with malaria parasites. With time, the malaria parasites forms daughter parasites and cells size grows with time. Figure 3a shows the AFM topography of the cell with no obvious surface indication of infection sites/parasites. Figure 3b clearly shows the remarkable example of how NFAH reveals daughter parasite nuclei embedded in RBC, not seen in normal AFM topography scans. Contrast in the NFAH image is due to time dependent phase delay of the acoustic waves reaching at the sample surface, which shows the change in the viscoelastic response. Given the operating frequencies, the sub-surface resolution of less than 10 nm can be achieved with this technique. This may open new vistas for in-vitro biological imaging, especially monitoring the nanoparticles tag pathways into cellular machinery.

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Figure 1:

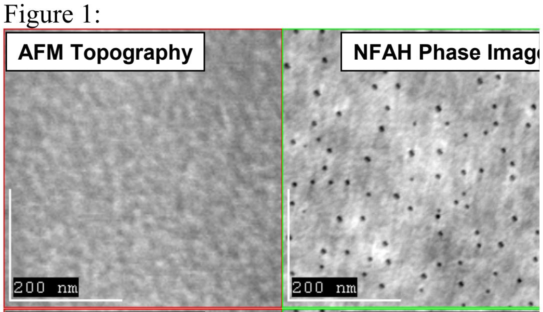

Figure 2:

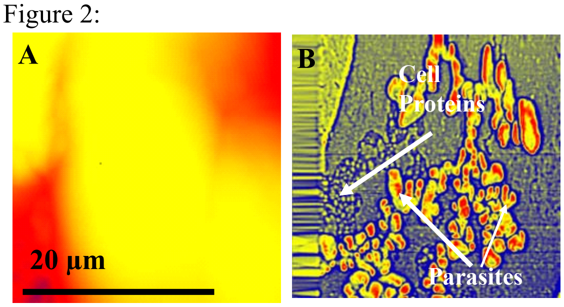

Figure 3:

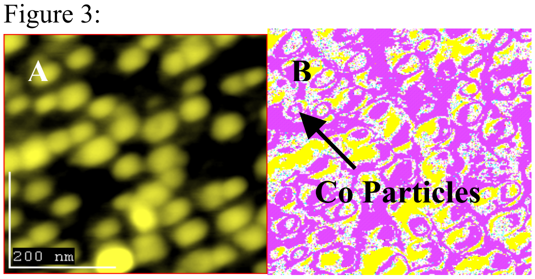

Figure Captions:

Fig. 1: (A) Topography (B) NFAH image clearly shows the sub-surface image of gold particles.

Fig. 2 (A) Topography scans of silica particles and (B) NFAH image clearly shows the sub-surface image of Co magnetic particles enclosed in silica.

Fig. 3: (A) 20µm x 20 µm topography scan shows couple of RBC cell structure. (B) NFAH image clearly shows the daughter parasites.

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