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Spectro-Nanoscopy of Ultrathin Films of Organic and Biological Specimens via Infrared Photo-induced Force Microscopy (IR PiFM)

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Ultrathin films of organic, inorganic, and biological molecules are utilized in many processes involved in production of nano-medicine, biosensors, and semiconductor devices. A non-invasive analytical technique that can verify the chemical and structural integrity of such thin films is presented in this paper. Infrared photo-induced force microscopy (IR PiFM) combines the two well-known techniques – atomic force microscopy (AFM for microscopy) and infrared absorption spectroscopy (IR for spectroscopy) – into one instrument to provide both structural imaging and IR spectro-nanoscopy with ~ 5 nm spatial resolution [1].

Three samples, two with ~30 nm thick peptoid film, each before and post annealing, and a monolayer film with less than 3 nm thickness, were prepared for IR PiFM analysis. Si substrates were cleaned with UV ozone, and the peptoid solution was spun coat to get ~ 30 nm thin films (sample 1: as-deposited thin film). The thin films were then annealed at 150 °C for 30 min (sample 2: annealed thin film). After annealing, the excess (i.e., ungrafted) peptoids were rinsed away by repeated sonication in a good solvent N-methyl pyrrolidinone and isopropanol, then annealed at 100 °C for 10 min to remove residue solvent (sample 3: monolayer).

On the thicker peptoid films, the IR signature associated with the peptoid film (acquired by FTIR) was clearly observed in PiF-IR spectra at every location while the spectral image at the amide I band (1633 cm^{-1}) showed a mostly uniform peptoid film. In addition to the IR peaks associated with the peptoid, additional peaks associated with common airborne organic contaminants were observed as well. On the monolayer sample, spectral mapping at the amide I band and local PiF-IR spectra revealed that only nanometer-scale fragments of peptoid had managed to graft onto the Si substrate as shown in Figure 1. Detailed analysis of the PiF-IR spectra and PiFM images will highlight the exceptional spatial resolution and sensitivity of IR PiFM and its usefulness in the analysis of ultrathin films of organic and biological specimens.

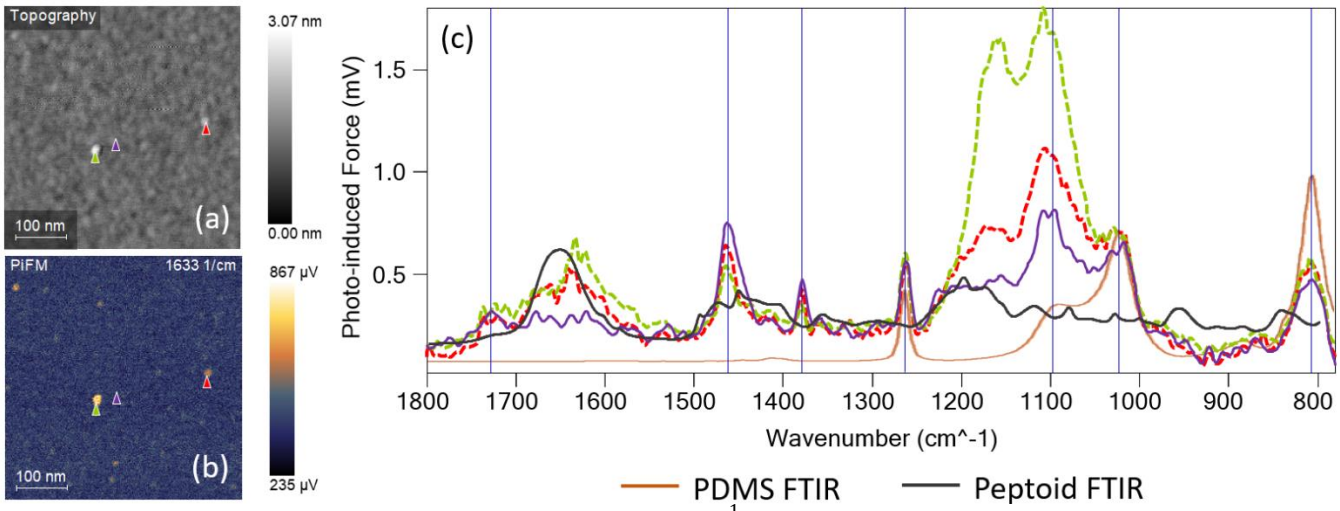

Figure 1. AFM topography (a), PiFM image at 1633 cm^{-1} (b), and PiF-IR spectra along with FTIR spectra for the peptoid and PDMS (c). The green and red dashed PiF-IR spectra are acquired on particles that are 1.8 nm and 1.2 nm tall, respectively, demonstrating the single-molecule-level sensitivity of PiFM. The PiFM image (b) shows that there are even smaller peptoid fragments remaining that are difficult to distinguish in topography due to the roughness of the substrate. The vertical lines denote the PiF-IR peaks that are attributable to contaminants.

References:

[1] D Nowak et al., Sci. Adv. **2** (2016), p. e150157.

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