

Nanoscale Organic Defect Characterization with AFM-IR

Curtis Marcott¹, Michael Lo², Qichi Hu², Kevin Kjoller² and Craig B. Prater²

¹ Light Light Solutions, Athens, GA, USA

² Anasys Instruments, Santa Barbara, CA, USA

The performance of many materials depends on their surface properties, composition, uniformity, and topography. Since the presence of small defects can have a dramatic effect on the ultimate properties of a material, it is important to have analytical tools capable of analyzing such defects and understanding their chemical makeup. Infrared (IR) spectroscopy is a powerful tool for obtaining chemical information related to a material. Unfortunately, the wavelength of light used to make the measurement limits the size of defects that can be reliably identified by IR spectroscopy. Diffraction typically limits the spatial resolution of IR microspectroscopy to 3-10 μm , making this technique problematic for identifying many material defects which can be much smaller than this. Atomic force microscopy (AFM), on the other hand, provides exquisite spatial resolution (as small as one nanometer), but this technique does not provide definitive information enabling the identification of the material or defect.

Recently, AFM and IR spectroscopy have been combined in a single instrument enabling the chemical identification of structures less than 100 nm in size [1-5]. In this paper, we describe a resonance enhanced infrared nanospectroscopy (REINS) approach that uses a rapidly pulsed quantum cascade laser (QCL) capable of repetition rates that can be tuned to the natural resonant frequencies of the AFM cantilever, thereby enhancing the sensitivity of the technique by about two orders of magnitude [6-7].

Figure 1 shows a diagram of the REINS experimental setup. The QCL repetition rate is tuned to match one of the natural resonance modes in the AFM cantilever, resulting in a signal enhancement over two orders-of-magnitude higher than the non-resonant experiment which typically employs a lower rep rate laser (i.e., OPO, or optical parametric oscillator).

Figure 2 shows the AFM topographical image of a flat metal substrate with a small defect in the middle of the field of view (shown in white). Just below the AFM image, a linescan (recorded along the horizontal red line in the AFM image) shows the height of the defect to be only 28 nm. The red IR spectrum shown on the right side of Fig. 2, collected at the AFM tip while it was in contact with the highest elevation point of the defect, clearly shows the presence of an organic material containing a carbonyl material (note band at 1740 cm^{-1}). The blue spectrum was recorded from a location further away from the defect. The REINS technique clearly shows great potential for assisting in the chemical identification of nanoscale organic defects [8].

References:

- [1] A. Dazzi *et al*, *Opt. Lett.* **30** (2005), p. 2388.
- [2] A. Dazzi *et al*, *Ultramicroscopy* **107** (2007), p. 1194.
- [3] A. Dazzi *et al*, *Ultramicroscopy* **108** (2008), p. 635.
- [4] C. Marcott *et al*, *Appl. Spectrosc.* **65** (2011), p. 1145.
- [5] A. Dazzi *et al*, *Appl. Spectrosc.* **66**, (2012), p. 1365.
- [6] F. Lu and M. Belkin, *Opt. Exp.* **19** (2011), p. 19946.

[7] F. Lu *et al*, Nat. Photonics, in press. doi:10.1038/nphoton.2013.373

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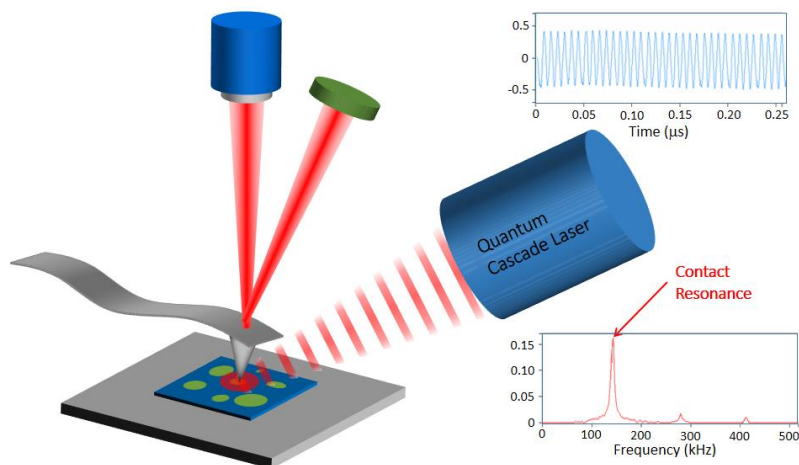


Figure 1. Diagram of the resonance enhanced AFM-IR experimental setup (left). A rapidly pulsed QCL creates a thermal wave in sample when tuned to an absorbing wavelength in the sample, whose expansion is detected using a cantilever deflection laser system. Time domain (top) and frequency domain (bottom) AFM cantilever deflection signals are shown on the right.

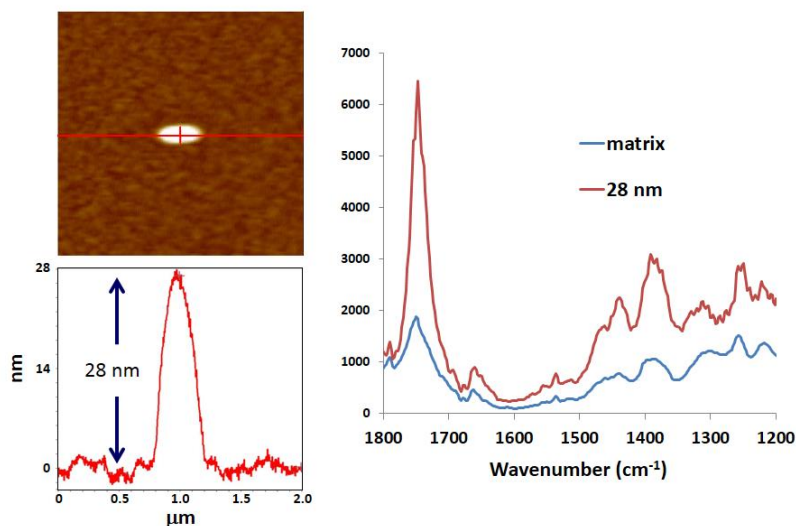


Figure 2. AFM topography (top) and linescan (bottom) showing a white defect area in the center of the 2- μm x 2- μm AFM image recorded on a metal substrate. The height of the defect is 28 nm. Spectra shown on the right were recorded with the AFM tip centered in the white defect area of the image (red spectrum) and in the surrounding matrix (blue spectrum).