Stability of AFM probes during prolonged PTIR measurements

INTRODUCTION

Mid-infrared (IR) spectroscopy allows for non-destructive and label-free analysis providing molecular specific information. IR spectroscopy has a certain limitation: the spatial resolution of IR microscopic imaging cannot be better than a few micrometers due to the diffraction limit in far-field microscopy. Photothermal induced resonance (PTIR) is a technique that allows IR spectroscopic and chemical image analysis at the nanoscale. PTIR make use of the sensitivity and the high resolution of the atomic force microscope (AFM) to sense a special interaction between a sharp tip and the sample. AFM is used to detect IR absorption of a sample by means of sensing its photothermal expansion upon repeated excitation. By raster scanning the sample, which is illuminated with a wavelength of interest, and collecting the signal from every point of measurement chemical maps can be generated.

PTIR technique is capable of qualitative and quantitative material characterization. For that the PTIR system needs to deliver reliable and reproducible results. The stability of the system depends mainly on the stability of its key elements: mid-IR laser (power stability and pointing stability), AFM and an AFM probe (tip). Additionally, the performance of the system strongly depends on the method of the PTIR signal detection. Besides that, the stability of measurements is influenced by the external factors such as mechanical vibrations and atmospheric factors (e.g. water vapor). Aging of AFM probes during the measurements was not considered so far. In work we investigate for the first time the influence of the AFM probe stability on the performance of the PTIR system.

A degraded (aged or contaminated) AFM tip might significantly reduce the sensitivity of the PTIR measurements. Since the reduced or vanished PTIR signal leads to false quantitative analysis or poor quality chemical images. It is important to define when the probe starts degrading. When it comes to an investigation of an unknown sample it is difficult to distinguish whether the PTIR signal is small due to the low absorption in the sample (low concentration of an analyte) or due to the degraded tip.

RESULTS

We observed a silicon band related to the AFM tip in the PTIR spectra. We attribute this band to the tip contamination with PDMs (present in packaging materials). For the first time we performed prolonged measurements of a polymer and concluded that the tip degradation does not occur after extensive measurements; we found that the AFM tip performance might get worse after several measurements of biological samples and proposed a method for AFM tip quality control; we suggested to use $6\text{S}(-\text{Si-CH$_3$})$ band at 1260 cm$^{-1}$ to monitor the aging of a tip. With this method tip degradation can be evaluated in situ while measuring the PTIR spectra at the gold surface. A tip would be considered contaminated when the signal intensity of the silicon band is reduced or disappeared; we supported our findings with SEM. SEM visualized the contaminations at the AFM tips that showed the reduced PTIR sensitivity (reduced signal).

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