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In Situ Frequency Measurement of Individual Nanostructures Using Fiber Optical Interferometry

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In this paper we describe a setup for the resonance frequency measurement of nanocantilevers, which displays both high spatial selectivity and sensitivity to specimen vibrations by utilizing a tapered uncoated fiber tip. The spatial selectivity is determined by the tip geometry, the high sensitivity to vibrations stems from interference of wave fronts reflected on the specimen and on the fiber tip itself. No reference plane on the specimen is needed, as demonstrated with the example of a freestanding silicon nitride cantilever. The resulting system is integrated in the DB-235 dual beam FIB system, thus allowing the measurement of sample responses in-situ, during observation in SEM mode. By combining optical interferometry and narrow band RF amplification and detection, we demonstrate an exceptional vibrational sensitivity at high spatial resolution.

PACs 7.60.Vg , 46.40-f

Measurement of resonance frequency, damping and stress related frequency shifts can be utilized to directly assess the mechanical ¹ and electrical ^{2,3} properties of nanostructures. This field is of increasing interest, because modern sensors and even complete analysis systems are integrated onto single chips (MEMS accelerometers and gyroscopes, Lab-On-A-Chip analyzers (a good review is found e.g. in ^{4,5,6}). These applications demand a thorough mechanical characterization of their constituents (e.g. valves, cantilevers and

springs) to predict resulting resonant frequencies and material strengths. Another direction is anticipated in the literature with the use of high-quality integrated resonators replacing the external crystals for clocked electronics or high quality on chip filters for RF applications. Measurement of the elastic properties is typically carried out using an excitation scheme which could be mechanical or electric field driven and a sensitive detector, using capacitive pickup or optical feedback.

To achieve lateral resolution in the subwavelength regime, tapered fibers are commonly used in scanning near field optical microscopy. Usually, a metal coating with a small aperture is brought onto the taper tip, allowing only evanescent waves to emerge, therefore circumventing the diffraction limit. Recently, it has been found ⁷ that spatial resolutions down to a fraction of a wavelength can also be obtained using uncoated fibers (collection mode scanning optical microscopy ⁸). The observed high degree of lateral resolution is explained by assuming that the probe tip acts as a spatially highly selective antenna, which determines the aperture for the backscattered light waves by its geometry. In this way, the irradiating spot, which is inevitably broadened by light leakage from the taper flanks, does not directly deteriorate the achievable resolution. Furthermore, the backscattered light interferes with the wave front reflected on the tip end face ⁹, which is utilized here for a sub wavelength sensitive sample vibration detection.

The integration of such a setup into a dual-beam FIB is relatively simple, using a purely fiber based setup. Once the optical components are plugged together, there is no further need for external adjustments; only the tip needs to be brought to the sample by a micromanipulator.

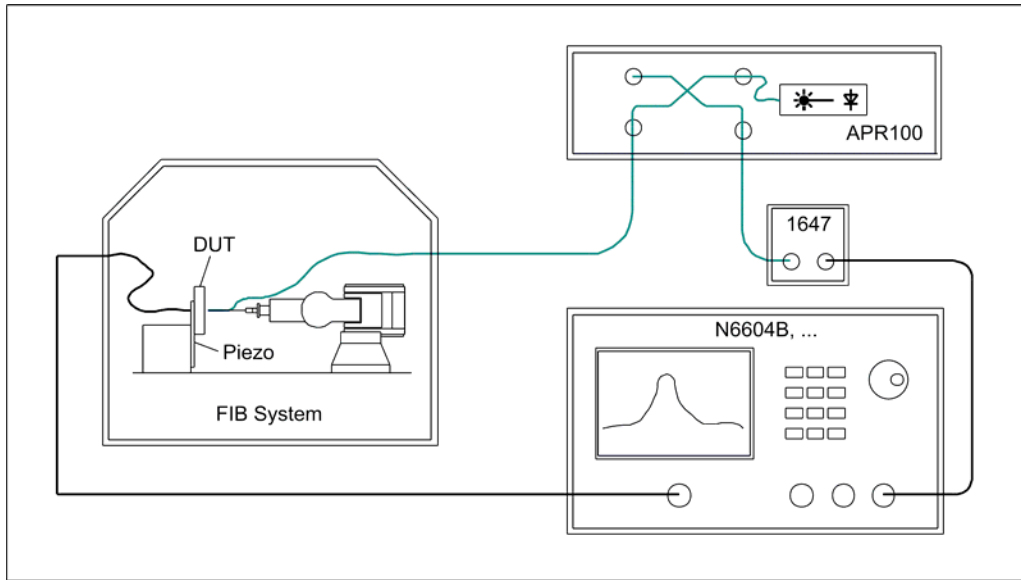


Figure 1: Schematic of experimental setup. Black lines: Coaxial cable; Blue lines: Optical fiber.

The optical system was built onto the base of an Attocube ARP-100 laser detector coupler. From the unit, the 2:2 fiber coupler and the infrared diode laser (wavelength 1310 nm, power on port 0.5mW) were used. As a detector we selected the New Focus Photodiode Detector model 1647. All components are plugged together using FC fiber connectors and feedthroughs.

The signal from the photo detector is fed into either a spectrum analyzer or a network analyzer. We have tested several units. The results discussed here were measured with a Agilent N9320A.

The optical fiber probe was formed using CO₂ laser heating, with the P-200 Sutter Instruments fiber puller[®]. To avoid charging, in the first experiments, the fiber was covered with colloidal silver or with aluminum film using physical vapor deposition

system. The tapered fiber was attached to a Kleindiek Micromanipulator MM3A-EM[®] for positioning.

The described system has been set up and used at the NCEM dual beam FIB. The specimen used to characterize the setup were chip mounted silicon nitride cantilevers with resonant frequencies ranging from 1 to 100 kHz. They were excited mechanically by means of piezo discs attached to a FIB sample holder. The S_3N_4 cantilevers are fabricated by the micromachining using optical lithography.^{10,11}

Prior to the experiments, the fiber end face was cut perpendicular to the fiber axis, to achieve a clean and optically transparent end face. For the first measurements, monofrequent excitation source was selected, using a Stanford Research DS345 signal generator, which allows also precise excitation amplitude control.

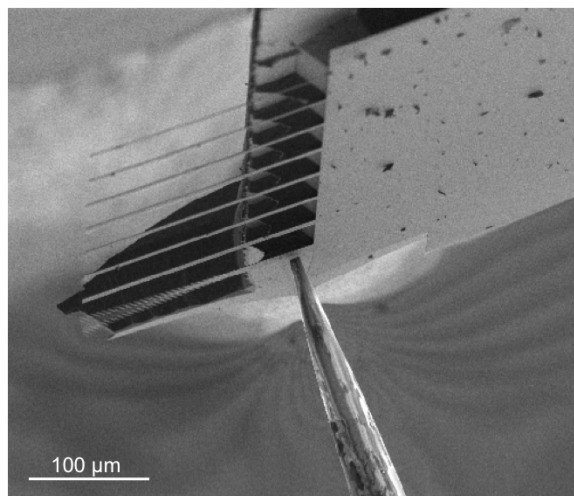


Figure 2: View of the tapered optical fiber approached to a nanocantilever.

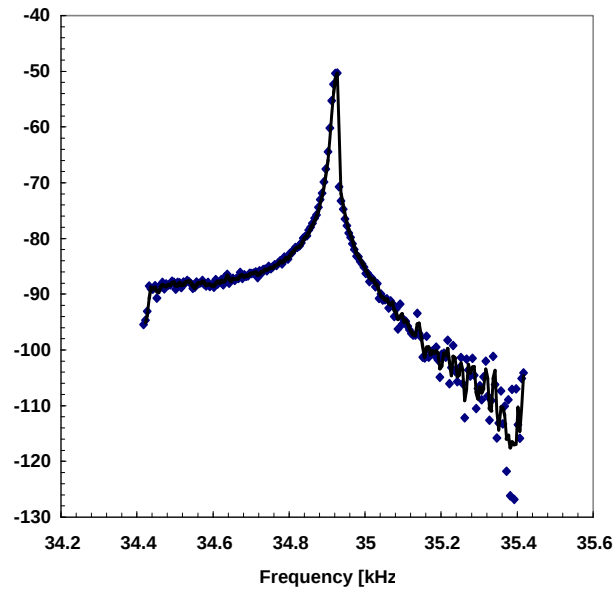


Figure 3: Signal from the arrangement in Fig. 2 . For this cantilever, the excitation source was a signal generator with a fixed frequency. The fiber had an end face diameter of ca. 1 μm .

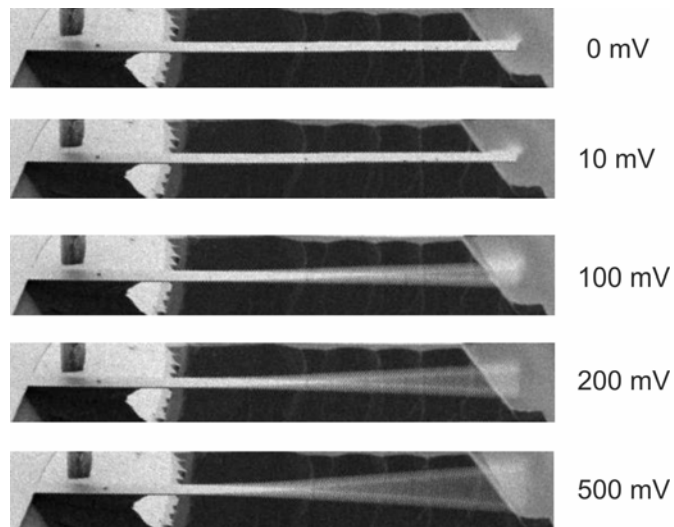


Figure 4: Nanocantilever at different excitation amplitudes. Above the support lamella on the left hand side, the optical fiber is in close distance to the surface

The first measurement discussed here was done on a part of the cantilever, which was supported by a silicon lamella and displayed no resonantly amplified vibrations during the measurement (Figure 4). Therefore, only the vibrations stemming from the piezo support are measured. The response curve is shown in Figure 5. The response signal amplitude calculated from the spectrum analyzer profiles scales surprisingly linear with the excitation amplitude. Note that in the SEM images, no vibration of the structure is yet detectable. The resolution of the SEM images was set to $0.45 \mu\text{m} / \text{pixel}$. So clearly, the probe is sensitive to vibrations well below the wavelength of the irradiating light.

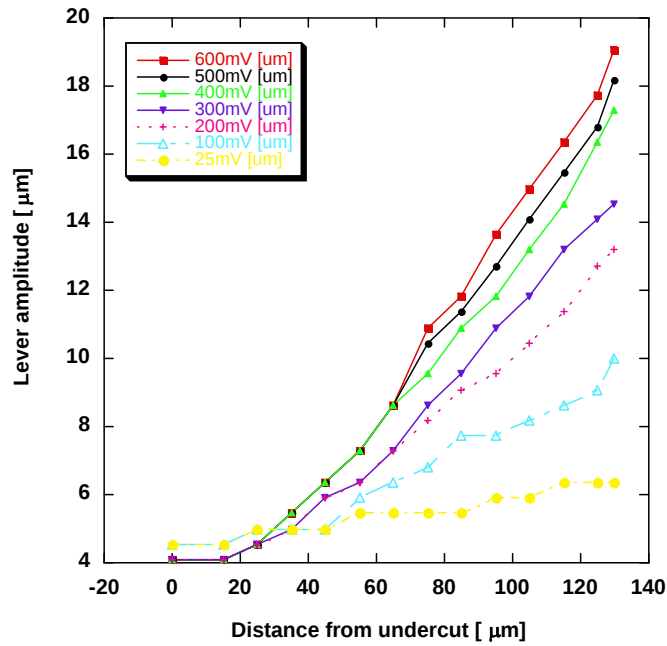


Figure 5: Detector response on different excitation amplitudes (a) and renormalize response curve (b) for the arrangement as shown in Fig. 4 .

In a second run, the vibration amplitude was measured along the cantilever. The resulting profile is shown in Figure 6 . The comparison to the images given in Figure 3 shows, that

the vibration amplitude in the region below 15 μm , which corresponds to the support by the lamella, is constant and increases as the free part of the beam is reached.

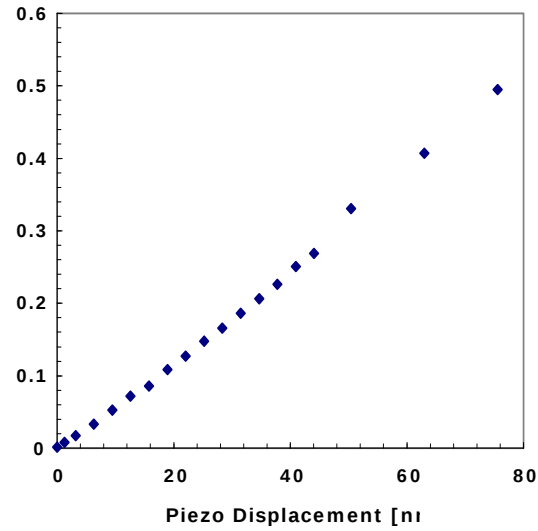


Figure 6: Measured detector output along the cantilever from Fig. 4. Between 0 and 15 μm , the cantilever is supported by the lamella below the cantilever.

In the third run the lateral position of the probe was moved across the sample to determine the lateral resolution of the probe. It is to be noted that for

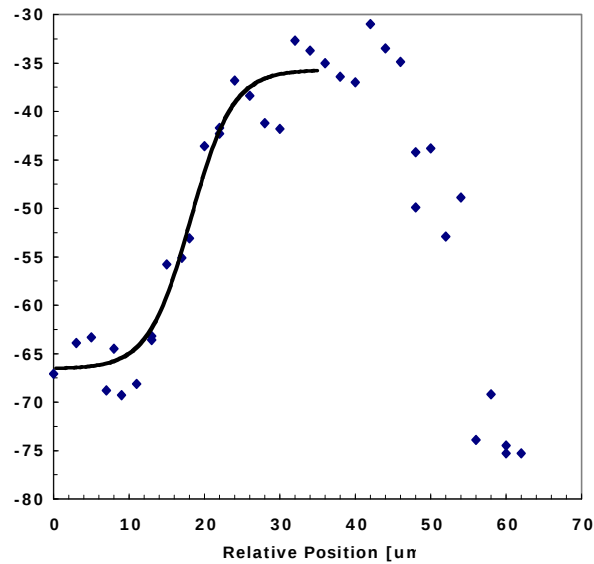


Figure 7: Measured profile across the cantilever from Fig. 3. The probe collects light at ca. 3.5 μm above the cantilever surface. The line is a fitted tangens hyperbolicus function.

specimen of the size similar to the wavelength, diffraction effects modify the amplitude of the backscattered intensity, which in turn modify the response. From the fitted tangens hyperbolicus function, a lateral resolution of 7.7 μm was extracted (20% - 80%).

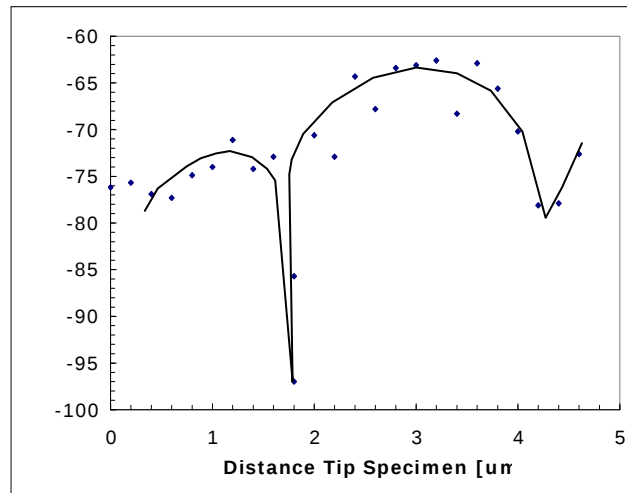


Figure 8: Detector response on variation of tip-specimen distance (the line is to guide the eye).

To characterize the measured signal sensitivity depending on the specimen distance, we varied the tip-specimen distance at constant excitation. The resulting curve is displayed in Fig. 8 . The curve shows a clear and reproducible dip at about 1.8 μm and had an overall maximum at 3.4 μm distance. Note that the distance was recalculated from the images, taking a sample tilt into account that was determined later on. The optimum can result from the overlay of several influences, namely the diffraction modifying the backscattered wave intensity distribution, the balance of the light wave amplitudes

backscattered from the specimen and from the probe tip end face (resulting in different fringe visibilities), and probably also parasitic interference stemming from the various FC connections in the fiber path. The dip could be the consequence of measuring at a fringe maximum, where vibrations at a frequency f would result in intensity variations of $2f$.

We have presented a useful approach for a versatile vibration measurement setup. It is rugged and needs, besides the tip positioning, no other adjustments. The high spatial selectivity of the device enabled the addressing of individual nanostructures and a very sensitive evaluation of the present vibrations. Through the combination of two sensitive acquisition methods, namely optical interferometry and narrow band RF amplification and detection, we were able to demonstrate an exceptional combination of spatial and vibrational sensitivity.

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