[1029] Mapping of vibrational modes of nanoscale membranes via scanning probe microscopy.

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The development of new miniature devices with actuation and/or detection of motion of their components, such as found in micro and nano-electromechanical systems (MEMS and NEMS), requires the development of appropriate nanoscale resolution techniques for their characterization. While the dynamic behaviour of MEMS and NEMS based on suspended membranes could be studied by optical methods, such as interferometry and Laser Doppler Vibrometry (LDV), spatial resolution of these techniques is inevitably limited by the light wavelength to the micrometre length scale. In this work, we show that SPM techniques that offer much higher spatial resolution down to few nanometres, can be effectively used to analyse the vibrations of a nanoscale thin membranes over the frequency range from few kHz to tens of MHz using both linear and nonlinear mechanisms for the excitation and detection of such vibrations.

As a model of MEMS system, we used a 200 nm thick Si_3N_4 membrane (Agar Scientific) with 500x500 um^2 size manufactured on the 400 um thick Si substrate. The Si substrate was glued to the ultrasonic transducers (Physik Instrumente, UK) capable of producing at up to 10 MHz vibrations normal to its surface.

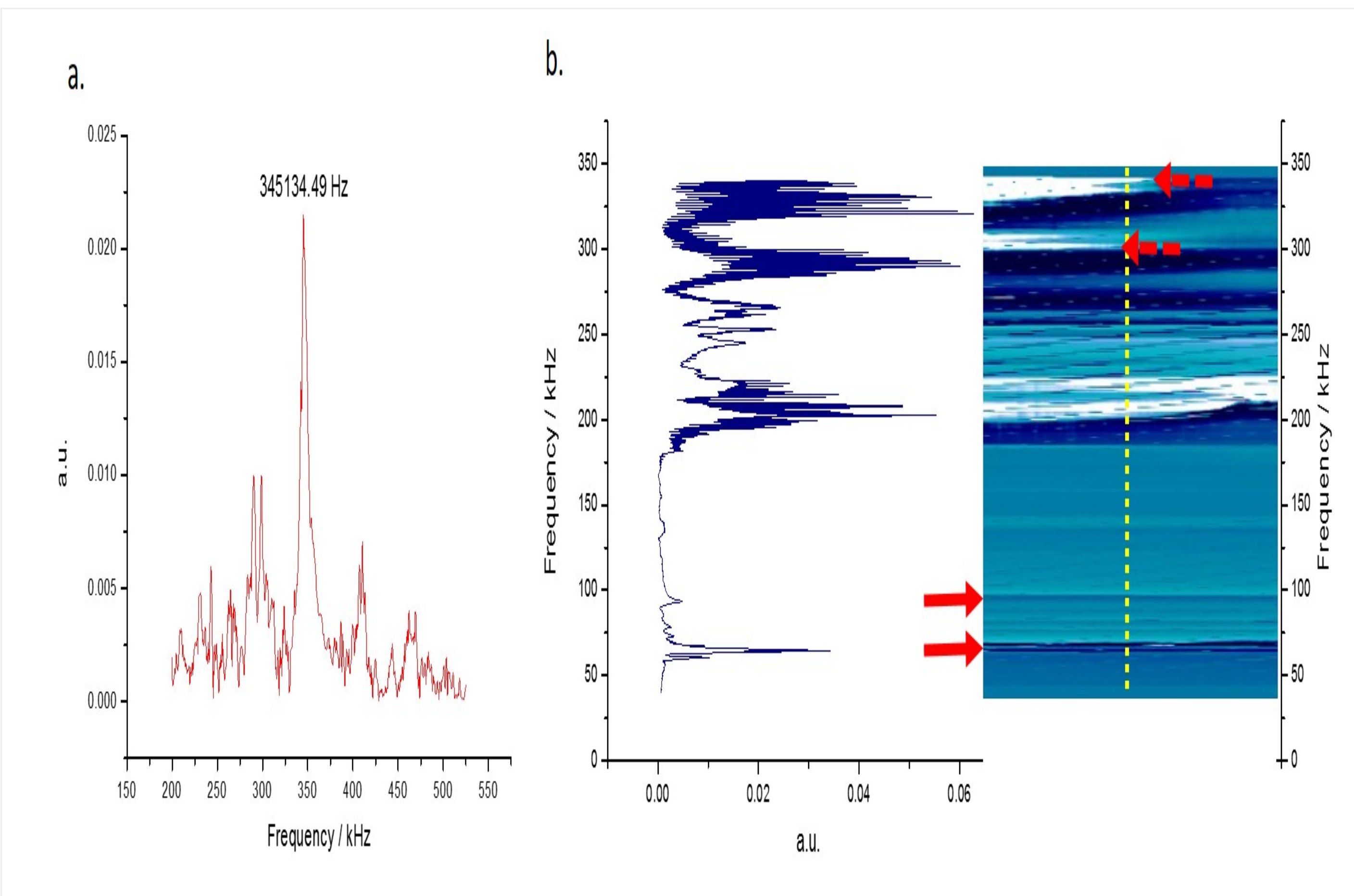
First SPM technique employed in this study, were based on the contact atomic force microscopy (AFM) (Nanoscope V with MultiMode scanner, Bruker) and used detection of the vibration on the excitation frequency f_m – approach known as force modulation microscopy (FMM) [1]. Second SPM method we used provided membrane excitation via high frequency (HF) carrier with frequency f_{HF} modulated at f_m with the detection at the modulation frequency – similar to the one used in the ultrasonic force microscopy (UFM) [2]. In order to effectively create high frequency modulation with f_m at few MHz range and above we used addition of two HF electrical signals at the adjacent frequencies f_1 and f_2 using a two-channel function generator (Rigol DG1062Z, frequency range up to 60 MHz). This effectively created an amplitude modulated waveform at f_{ex}=(f_1+f_2)/2 frequency modulated at f_m=f_2-f_1 frequency applied to the sample. The sync outputs at the f_1, f_2 frequencies were mixed by a separate double-balanced mixer (Hewlett Packard, HP 10534A) with output low-pass-filtered and amplified via wideband amplifier (TTi WA301).This created a reference signal at the modulation frequency for the HF lock-in amplifier (SRS 844, 200 MHz range, Stanford Research). We call this mode HF Modulated UFM (M-UFM). The response of the AFM deflection was collected via in-house made signal access module and detected via oscilloscope or the lock-in amplifier.

In order to interpret the FMM and M-UFM results, the vibration frequencies of the membrane and the AFM cantilevers, have been calculated using approach described elsewhere [3] with this calculation and AFM measurements compared with optical LDV measurements (deflection sensitivity of ±50 fm Hz$^{-1/2}$, 10 MHz frequency range, OFV-2570, Polytech). Both LDV, FMM and UFM measurements were controlled by the in-house developed LabVIEW software allowing complete control of the excitation frequencies and amplitudes and data collection.

LDV measurements allowed to clearly identify the cantilever flexural vibrational modes with their frequencies corresponding with the theoretical prediction within few percent accuracy. Furthermore, the obtained membrane frequencies of ca. 300 kHz (Fig. 1a) using in-plane membrane stress reported in the literature [3], allowed to confirm that the model of a tensioned membrane to be used, with the plate vibrational modes resulting in the frequencies of 5-10 kHz, several orders below the observed vibrational modes. During SPM measurements, we used UFM to locate the boundary between the membrane and the substrate, as the Si_3N_4 membrane topography was perfectly flat due to high in-plane tension, therefore not allowing to detect the edge between the free and supported membrane. We then performed a continuous scan along single line across the boundary of the membrane, while also simultaneously performing an excitation frequency sweep from 40 to 340 kHz. The 2D image (Fig. 1 b) shows the amplitude of the resulting cantilever vibration (image brightness) as a function of position of the tip across the edge (x-axis) and the excitation frequency f_m (y-axis). We observe that some of the vibration modes attributed to the contact cantilever resonances change the frequency and amplitude as the SPM tip moves to the suspended area of the membrane (solid arrows), while some attributed to the membrane modes appear only in the suspended membrane area (dashed arrows).

Finally, we tested the M-UFM mode that excites the membrane vibrations at the modulation frequency *f*m exclusively in the point of tip-membrane contact via nonlinear tip-surface excitation (as in UFM [2]). The resulting vibration is then obtained via linear detection of the probe – similar to the FMM. We have found that M-UFM excitation was indeed capable of exciting membrane resonances at frequency around $f_m=f_1-f_2$=245 kHz. With electrical drive amplitude of 5 V the ultrasonic vibration at f_1, f_2 ~ 4 MHz frequency was approximately 2 nm peak-to-peak.

In conclusion, we find that it is possible to reliably measure vibrations of the suspended nanoscale thin membranes using FMM and M-UFM approaches. The measured data were validated by the LDV measurements and theoretical analysis of the vibrational modes of the cantilever and the membrane. Both FMM and M-UFM potentially allow the spatial resolution of about 10 nm. With the demonstrated capability of UFM to work up to several 100 MHz frequencies, the M-UFM approach may be a useful addition for the exploration of the vibration of MEMS and NEMS structures of sub-um dimensions including ones based on the 2D materials.



(https://www5.shocklogic.com/Client_Data/RMS/al/MMC2017/upload/IRMS-MMC2017-37104651-Picture1.jpg)

Fig. 1 a) LDV spectrum of the membrane vibration. **b)** FMM of frequency sweep across the membrane edge, the brightness of the image reflects amplitude of the cantilever vibration, with x-axis - position of the tip across the membrane edge and y-axis the excitation frequency f_m.