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MEMS-SPM for dynamic nanomechanical measurements of soft materials

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Summary:

High throughput nanomechanical characterization of soft materials including biomaterials demands further development of advanced nanomechanical measurement systems. A microelectromechanical system based scanning probe microscope (MEMS-SPM) for nanomaterial testing has been developed to bridge the metrological gap between typical nanoindentation instruments and nanomechanical AFMs. Proof-of-principle measurements of typical reference materials including PC with a bandwidth up to 20 kHz have been reported to validate the advanced measurement capability of the MEMS-SPM.

Keywords: Nanomechanical measurements, nanoindentation, nano-dynamic mechanical analysis (nano-DMA), microelectromechanical systems (MEMS), scanning probe microscopy (SPM)

Motivation

Quantitative measuring the mechanical properties of soft materials is essential in the field of biomedical applications such as tissue engineering and regenerative medicine, material development and quality control for soft robotics including soft robotic actuators and components, biophysics and cell mechanics.

To date, nanoindentation instruments with diamond Berkovich and ball-shaped indenters have proven to be qualified for mechanical measurements of bulk materials. However, their relatively poor force resolution has limited the application of nanoindentation instruments for biomaterial measurements. Nanomechanical AFMs have become powerful tools for biomaterial testing with relatively high lateral resolution. However, the indentation force and depth of AFMs are quite often inadequate for quality control of hybrid materials.

To bridge the metrological gap between nanoindentation instruments and nanomechanical AFMs, recently a MEMS-SPM has been proposed [1]. Quasi-static measurements of typical soft materials including various thermal plastic polymers with the MEMS-SPM have demonstrated the fundamental performance of the MEMS-SPM for nanomechanical measurements.

In this manuscript, the MEMS-SPM system has been further developed for the purpose of high-throughput nanomechanical measurements of soft materials.

Principle

The schematic of the MEMS-SPM for high-throughput nanomechanical measurements is illustrated in Fig. 1. It consists typically of a group of electrostatic comb-drives for force modulation and indentation depth sensing, and a passive cantilever gripper integrated on the main shaft of the MEMS-SPM. This gripper is especially optimized to hold non-contact AFM probes for material testing.

With this integrated cantilever gripper, nearly all commercially available AFM probes with a tip height larger than ~ 5 µm can be clamped by the MEMS-SPM head and utilized for material testing. For nanomechanical measurements of extremely soft biomaterials, ball-shaped AFM probes are recommended in [1].

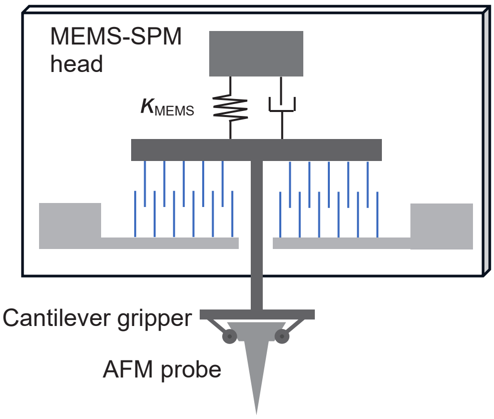

Fig. 1. Schematic of the MEMS-SPM head for nanomechanical characterisation of nanostructured materials.

In the case of quasi-static nanomechanical measurements, various capacitive sensors including commercial capacitance to digital converters (e.g. AD7745/46/47) can be utilized for indentation depth measurements with a resolution down to 10 Picometer.

To implement dynamic mechanical measurement methodology [2], a measurement system on basis of typical lock-in technique has been developed. As illustrated in Fig. 2, the carrier signal f_c overlapped with the modulation signal f_m is sent to the comb-drives within the MEMS for force modulation and indentation depth measurement. The through-put current coming from the MEMS is firstly converted to voltage signal and then sent into the lock-in amplifier for further analysis.

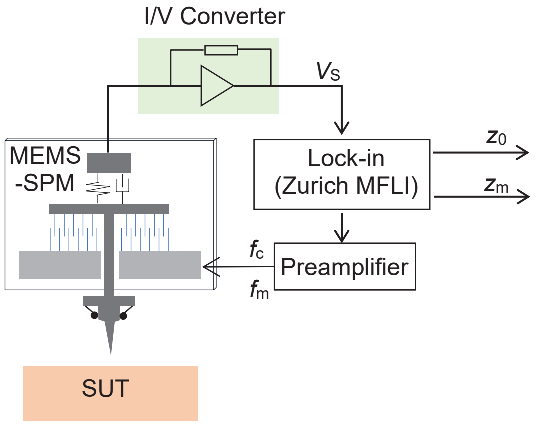

Fig. 2. Schematic of the signal processing and data acquisition system for dynamic mechanical measurement with the MEMS-SPM.

In our measurement system for dynamic measurements, the typical carrier signal f_c is set to 500 kHz for quasi-static indentation depth (z_0) measurement with subnanometric resolution and a bandwidth up to 300 Hz. The self-developed preamplifier for dynamic measurements features a DMA bandwidth up to 50 kHz.

Results

To demonstrate the capability of the MEMS-SPM for nano-DMA measurements, typical reference materials have been measured. Fig. 3 shows typical frequency-amplitude response of PC measured by the MEMS-SPM with a clamped diamond AFM probe. It can be seen that the MEMS-SPM head has a resonance frequency of about 2.9 kHz. After engagement with the reference sample, the contact resonance frequency of the measurement system is shifted to about 12.5 kHz.

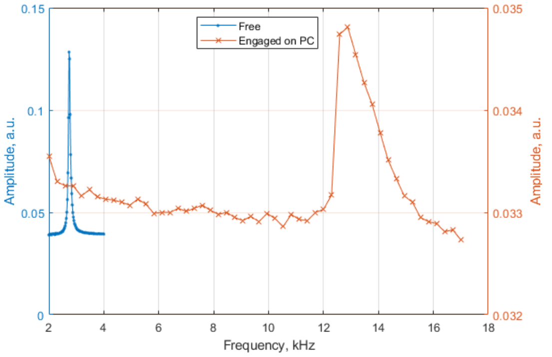

Fig. 3. Typical dynamic response of reference PC under MEMS-SPM measurements.

Summary and outlook

Quantitative data evaluation algorithm for MEMS-SPM will be further developed to extract the dynamic mechanical properties of soft materials including complex modulus and the loss factor. Quasi-static and dynamic nanomechanical measurements of the reference materials using commercial nanoindentation instrument (e.g. Hysitron Tiboindenter TI-950) will be performed.

The measurement uncertainty of the MEMS-SPM will be evaluated by means of the comparison measurements.

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