Draft Good Practice Guide for the measurement of the cantilever normal spring constant using a MEMS device

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Introduction

The methods for characterization of the normal stiffness of an AFM-cantilever can be classified as dimensional, dynamic experimental methods and static experimental methods, in which the static experimental method measures the deflection of a cantilever under static loading, so as to obtain the stiffness of a cantilever without any need of the prior knowledge of the cantilever. For this purpose a Micro-Electro-mechanical-System (MEMS) based active reference spring calibration system has been proposed: On basis of a MEMS nano-force transducer [1] with well-calibrated spring constant, AFM cantilevers with stiffness down to 0.02 N/m can be characterised with a measurement uncertainty below 5%. Details of the procedure are given in the following as guide for typical AFM users.

In the first part we will describe details of the calibration tool, of the hardware needed and of the software used, of the stiffness calibration of the MEMS itself. In the second part we describe the AFM cantilever calibration method and the corresponding data processing method.

1. Description of the MEMS nano-force transducer

1.1 Principle of the MEMS nano-force transducer

The MEMS-based nano-force transducer is developed based on the principle of lateral electrostatic comb-drive. As shown in Fig.1, the electrostatic comb-drive actuator consists of two interdigitated finger structures, in which one is fixed and the other one is movable. The force generated by N comb-pairs during electrical actuation is [2]:

$$f_e \approx N \cdot \varepsilon_0 \cdot \frac{h}{d} \cdot U^2 \tag{1}$$

in which $\varepsilon_0 = 8.85 \times 10^{-12} Fm^{-1}$ is the permittivity constant, *N* is the number of finger pairs, *h* is the height of the comb and *d* is the lateral gap between the movable and the fixed combs. *U* is the drive voltage applied between the movable and fixed fingers.

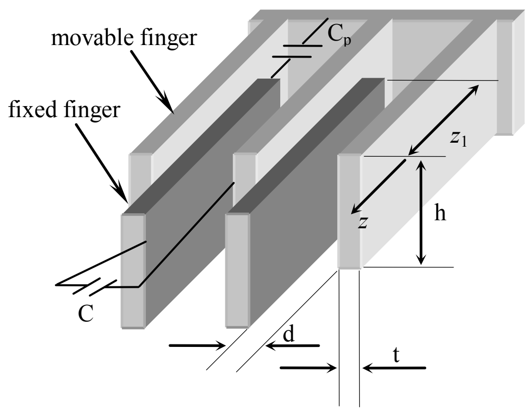

Fig. 1: Principle of the lateral comb-drive

The displacement of the movable fingers can be obtained by detecting the capacitance change ΔC between the movable and the fixed fingers.

$$\Delta C = N \cdot \frac{2\varepsilon_0 \cdot h}{d} \cdot \Delta z \tag{2}$$

1.2 Prototype of the MEMS nano-force transducer

The schematic of the MEMS nano-force transducer is shown in Fig. 2. The transducer has a left-right

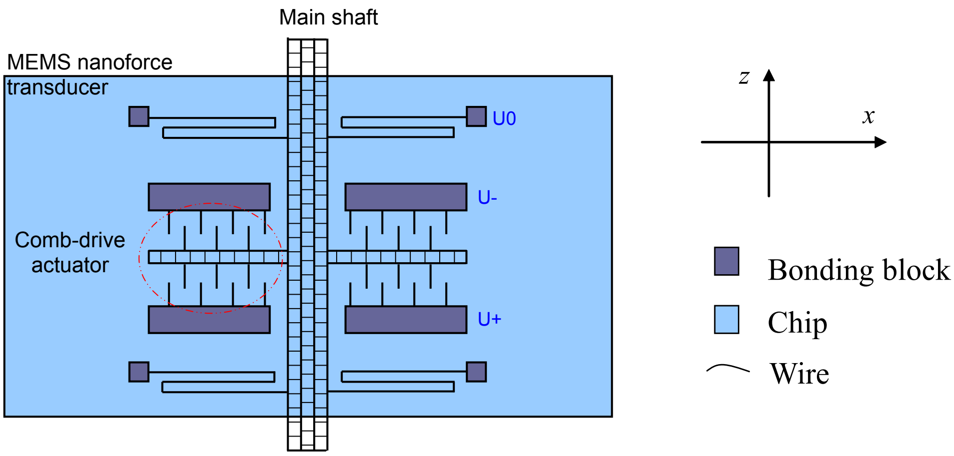

Fig. 2: Schematic of the MEMS nanoforce transducer

symetric structure, in which the movable part (main shaft) is suspended by four folded springs.

All the movable fingers are mounted on the beams coming from the main shaft. The main shaft is electrically connected to the electrode U_0 = 0 V and the fixed fingers are connected to the electrodes $U+$ (and $U-$). This voltage can be used to move the MEMS main shaft actively by 10 µm [3].

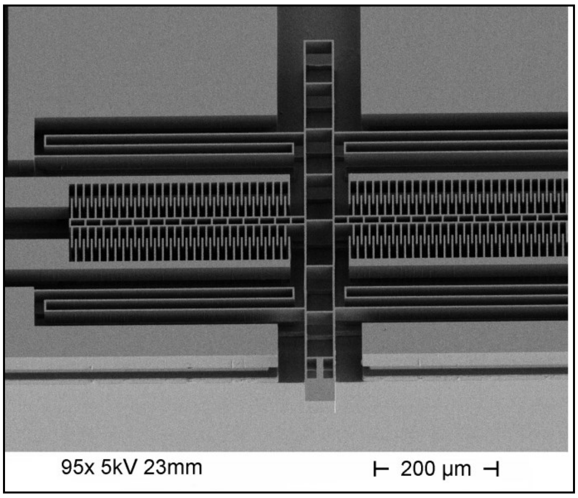

Fig. 3: Scanning electron microscope image of a MEMS nano-force transducer

1.3 Hardware for the MEMS calibration system

The hardware for the MEMS control and sensing system consists of the electronics, a power supply, a lock-in amplifier and a data acquisition board (NI USB6251) (see Fig. 4).

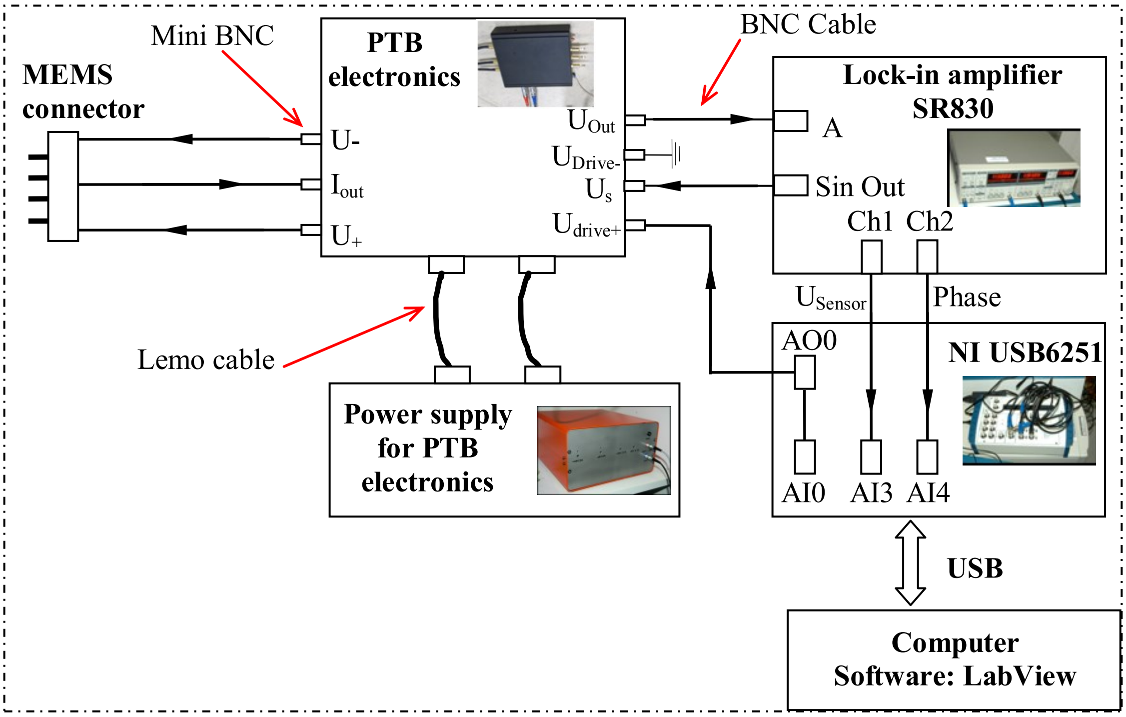

Fig. 4: Components and cable connections of the MEMS drive and sensing system

By measuring the capacitance variation due to the displacement of the main shaft of the MEMS force transducer the capacitive read out system reaches a sub-nanometer resolution.

A DC driving signal (U_{drive+}) (see Fig. 5) from 0~10 V can be added to a 60 kHz sinusoidal signal (Us). It is then amplified and applied to the U+ connector of the MEMS device. At the same time, the Us signal will be reversed and send to the other fixed part of the MEMS (U-). The capacitive output current I_{out} is converted to the detecting voltage U_{out} by an I/U converter, and finally demodulated by a lock-in amplifier (SR830, Standford Research Systems, Inc.).

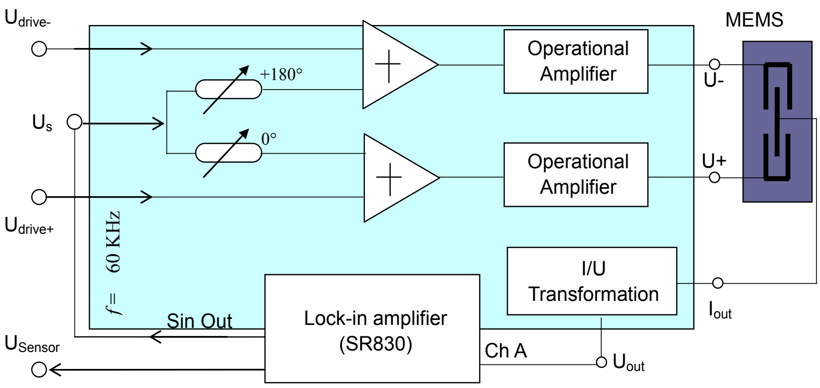

Fig. 5: Schematic and photo of the capacitive readout system of the MEMS reference spring

1.4 Software

LabView is used for programming the user interface and the data acquisition. The user interface for driving the MEMS and to obtain the measurement data is shown in Fig. 6.

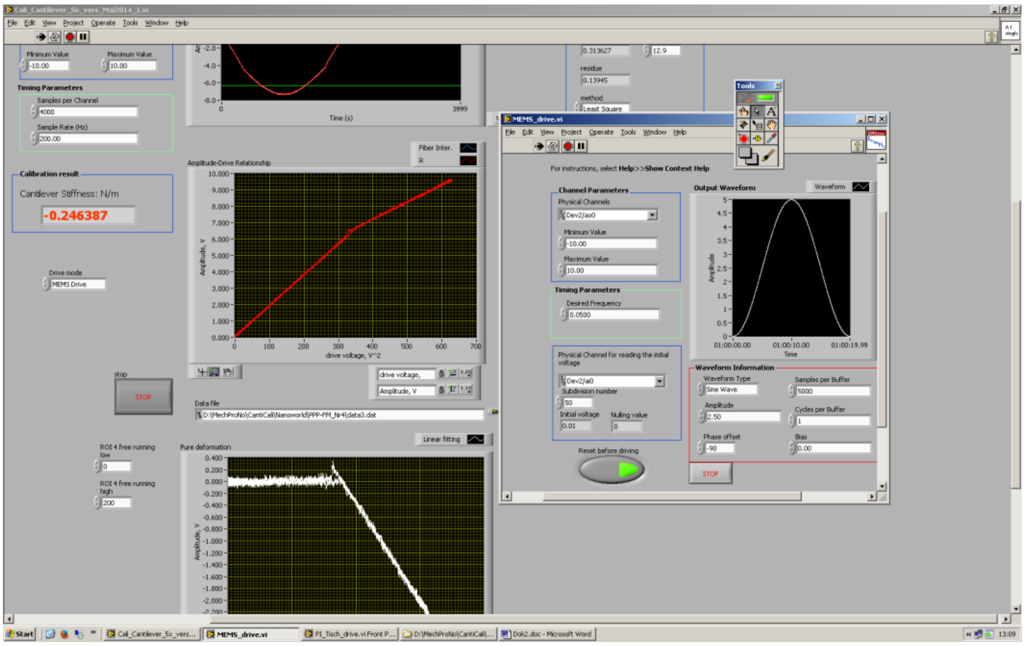

Fig. 6: User interface for the MEMS normal stiffness calibration system

During the measurement, temperature and humidity have to be measured.

2. Measurement of the cantilever normal spring constant using the MEMS force transducer

2.1 The stiffness calibration of the MEMS nano-force transducer

The stiffness of the MEMS nano force transducer k_{MEMS} needs to be firstly calibrated. It can be calibrated traceable with an uncertainty of 4 % [4] by using a precision compensation balance and a nano-positioning device to engage the MEMS to the balance (see Fig. 7) [5].

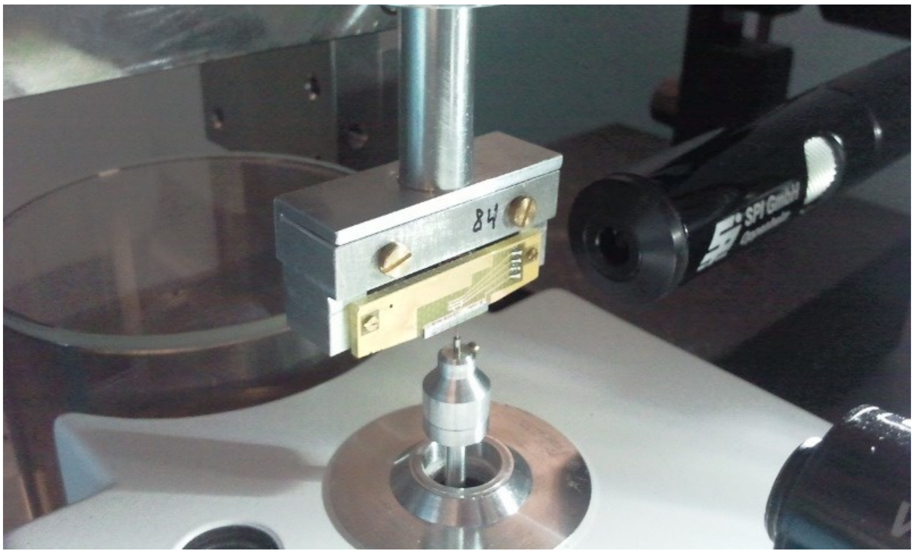

Fig. 7: Traceable MEMS stiffness calibration system

2.2 Applying the MEMS method for the stiffness calibration of AFM cantilevers

The schematic of the active reference spring method for cantilever normal stiffness calibration using a MEMS nano-force transducer is shown in Fig. 8. The test force F_t, generated by the nano-force transducer, lifts the main shaft of the MEMS until it gets into contact with the cantilever. Before contact, the slope of the force-displacement curve of the MEMS corresponds to the stiffness k_{MEMS} of

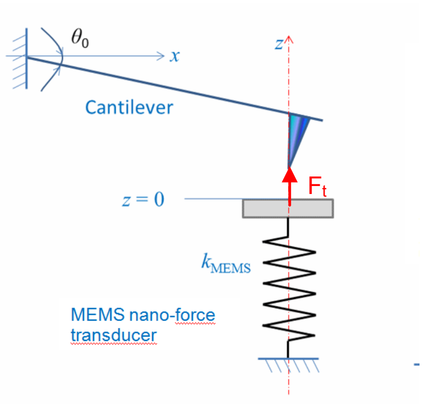

Fig. 8 (a): Schematic of the active reference spring method

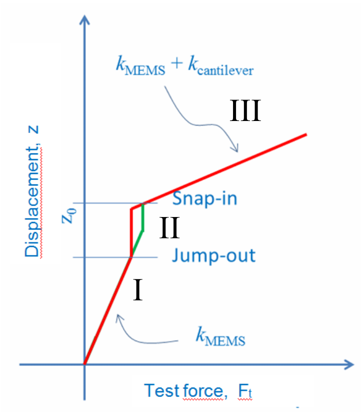

Fig. 8 (b): Typical calibration curve

the MEMS, as shown in Fig. 8(b), Phase I. After the MEMS getting into rigid contact to the cantilever, the slope of the force displacement curve changes due to the combined stiffness of the MEMS and the cantilever as shown in the Fig. 8(b), Phase III. Comparing the slopes of Phase I and Phase III, the normal stiffness of the cantilever can be calculated.

Since $F_t - k_{MEMS} \cdot \Delta z = k_{Cantilever} \cdot \Delta z$ in which Δz is the relative deflection of the cantilever-MEMS system, we have the combined stiffness.

$$k_{combined} = k_{MEMS} + k_{Cantilever} \tag{3}$$

Fig. 9 shows the experimental setup for the cantilever normal stiffness calibration using the MEMS nano-force transducer. The cantilever holder is fixed under at the objective (20x) of a microscope, and the MEMS is mounted on the specimen table of the microscope.

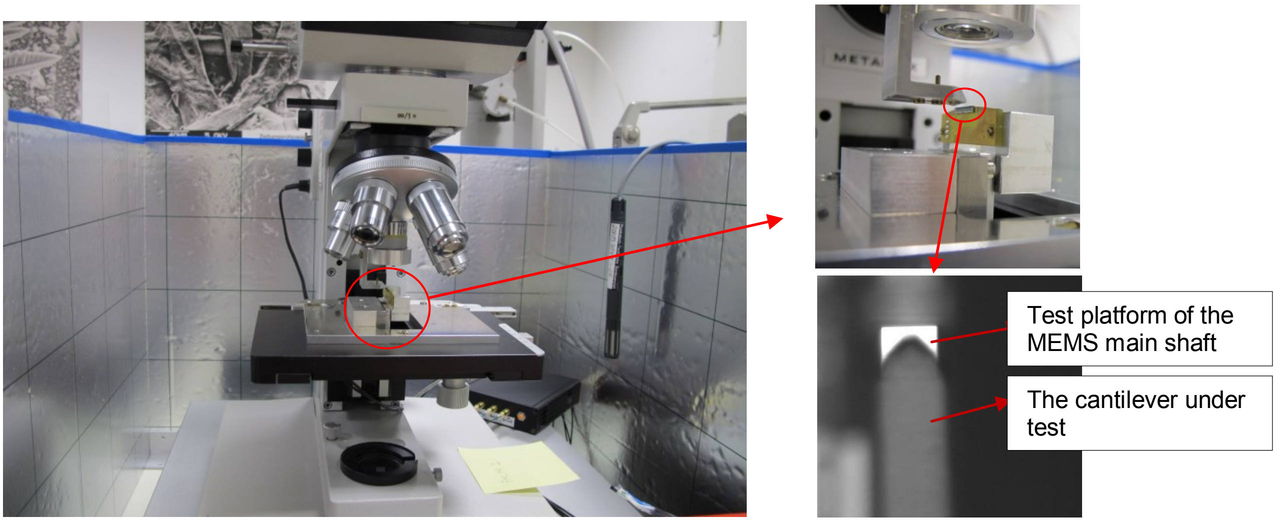

Fig. 9: Experimental setup for cantilever calibration using MEMS

Using the microscope the MEMS and the cantilever can be easily observed and aligned (see Fig. 9, bottom right).

Alternatively the MEMS can also be mounted directly under an AFM for onsite cantilever calibration. Fig. 10 shows one example in which the MEMS was mounted under the cantilever of a Digital Instruments AFM.

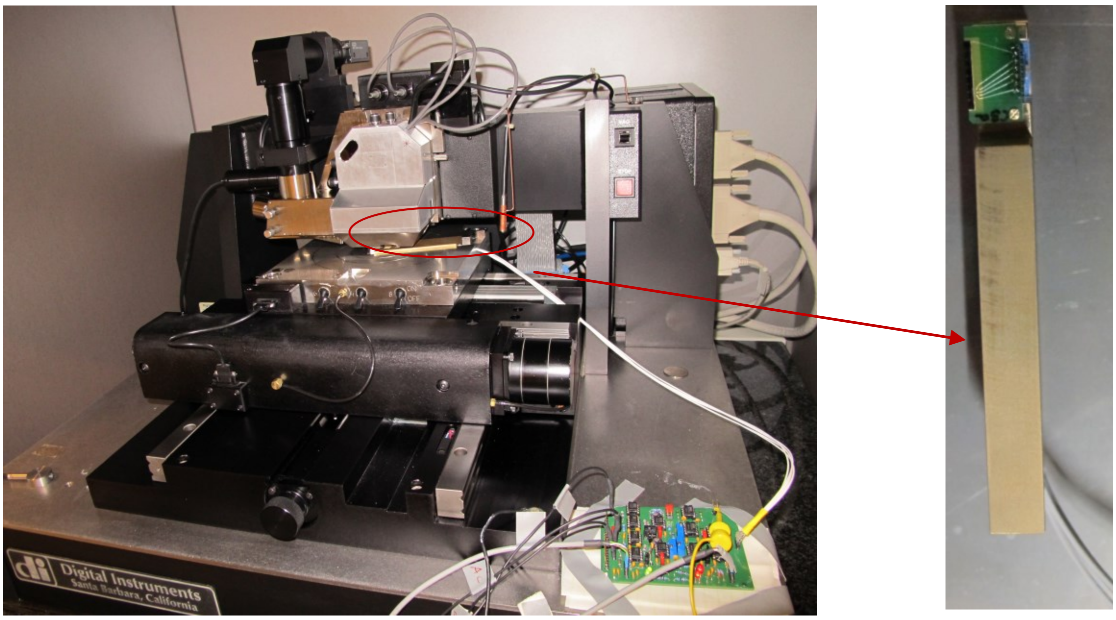

Fig. 10: Experimental setup for on-site cantilever stiffness calibration using a MEMS

A typical experimental calibration curve is shown in Fig. 11. Here the displacement of the MEMS Δz is detected by the capacitive measurement system of the MEMS (U_{sensor}), and the test force ΔF is proportional to the square of the applied voltage ($\sim U_{drive}^2$) between the fixed and movable fingers of the MEMS.

In Fig. 11 k_1 and k_2 are defined as the slopes of the $U_{sensor} \sim U_{drive}^2$ curve ($\Delta z \sim \Delta F$) measured by the MEMS nano-force transducer during loading and during unloading. Thus these slopes are inversely proportional to the stiffnesses k_{MEMS} and $k_{combined}$. The slopes have to be determined outside the regions of 'jump-in' and 'jump-out' [6].

From Eq. (3) it follows

$$\frac{k_{combined}}{k_{MEMS}} = \frac{k_1}{k_2} = \left(\frac{k_{Cantilever}}{k_{MEMS}} + 1\right) \tag{4}$$

Therefore we have

$$k_{Cantilever} = \left(\frac{k_1}{k_2} - 1\right) \times k_{MEMS} \tag{5}$$

The curve parts between points 0 and 1 for determining k_1 and the curve parts between the points 3 and 4 for determining k_2 need to be long enough and need to be of equal length for precise measurements.

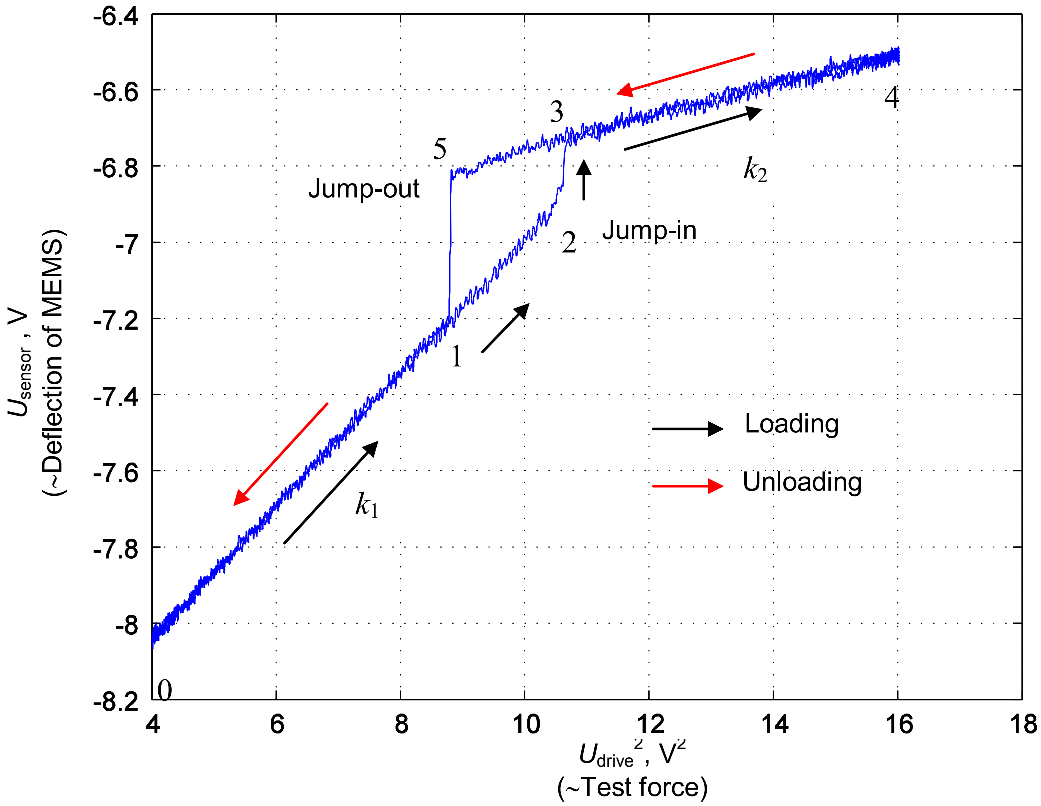

Fig. 11 Typical measurement curve for cantilever calibration with a MEMS device. A complete loading and unloading test round starts from the point 0 to 1→2 →3→4→3→5→1, and finally returns to 0.

Typical measurement times for one calibration curve consisting of a loading and a successive unloading cycle are 20 s.

At least 100 measurements have to be taken in order to assure a sufficient number of data points for calculation of mean values.

In order to average out drift effects the average cantilever stiffness is calculated as the mean of the loading and unloading values:

$$k_{\mathrm{Cant,mean}} = (k_{\mathrm{Cant,load}} + k_{\mathrm{Cant,unload}})/2 \tag{6}$$

From Eq.(5), the uncertainty for measuring the stiffness of the cantilever can be deduced as follows:

$$u_C^{\ 2}(k_{cantilever}) = \left(\frac{k_2}{k_1} - 1\right)^2 \cdot u^2(k_{MEMS}) + \frac{k^2{}_{MEMS}}{k_1^{\ 2}} \cdot u^2(k_2) + \frac{k^2{}_{MEMS} \cdot k_2^{\ 2}}{k_1^{\ 4}} \cdot u^2(k_1) \tag{7}$$

And the relative measurement uncertainty is expressed as follows:

$$\frac{u_C^{\ 2}(k_{cantilever})}{k^2{}_{cantilever}} = \frac{u^2(k_{MEMS})}{k^2{}_{MEMS}} + \frac{1}{\left(1 - \frac{k_1}{k_2}\right)^2} \cdot \frac{u^2(k_2)}{k_2^{\ 2}} + \frac{1}{\left(1 - \frac{k_1}{k_2}\right)^2} \cdot \frac{u^2(k_1)}{k_1^{\ 2}} \tag{8}$$

For simplification, the relative uncertainties for the determination of k_1 and k_2 can be considered to be the same, and equal to the relative stability of the self-calibration procedure, i.e. $\frac{u(k_1)}{k_1} = \frac{u(k_2)}{k_2} \approx 3 \times 10^{-4}$. When the cantilever's stiffness is far larger than that of the MEMS (i.e. $k_1/k_2 << 1$ we can estimate that the relative uncertainty for determining the stiffness of a cantilever, is close to that of the MEMS stiffness, i.e. $\frac{u(k_{MEMS})}{k_{MEMS}} = 4\%$ [4].

Further uncertainty contributions are the scattering of the measurement values estimated at 3 % and drift effects which are estimated with a contribution of 5 %. For cantilevers with stiffness deviating not more than a factor of ten from the stiffness of the MEMS thus a final stiffness uncertainty of 7 % results.

Literature

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