A New High Precision Procedure for AFM Probe Spring Constant Measurement using a Microfabricated Calibrated Reference Cantilever Array (CRCA)

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ABSTRACT

The accurate measurement of nano and pico-scale forces using Atomic Force Microscopy (AFM) is predicated on an accurate determination of AFM cantilever probe spring constants. This paper introduces a new calibration procedure that allows for high precision normal spring constant measurements of AFM cantilever probes using a unique microfabricated device called the Calibrated Reference Cantilever Array (CRCA).

Keywords: atomic force microscope, cantilever, stiffness calibration

1 INTRODUCTION

Atomic Force Microscopy (AFM) is used for a variety of applications in nano and biotechnology, including the accurate measurement of nano and pico-scale forces. However, accurate force measurement using AFM relies on an accurate determination of the spring constant for the AFM cantilever probe used.

A number of techniques have been proposed and refined to estimate AFM cantilever probe normal spring constants[1-11]. Existing calibration methods can be broadly categorized into either static or dynamic response techniques. Examples of dynamic calibration methods include the so-called Cleveland added mass calibration[1,9,10], which involves observing the resonance frequency of the cantilever before and after the placement of a mass onto its free end. The thermal method[2,4,8] assumes the cantilever can be modeled as a simple harmonic oscillator, and using the equipartition theorem, matches the normal spring constant of the cantilever with the ratio of thermal energy to the mean square of thermal fluctuations in the cantilever. The so-called Sader method[7,10] relates the normal spring constant of a rectangular cantilever to a function of its unloaded resonant frequency, quality factor (of that resonance) and dimensional properties in a well characterized fluid medium (usually air).

The most commonly used static (deflection) calibration approach has been the so-called Reference Artifact method, where deflection of the AFM cantilever of unknown stiffness is monitored as it is pressed against a calibration artifact of ‘known’ stiffness[3,5,6,8,11]. The method is suitable for a range of AFM cantilever probe types as well as shapes, such as those with metallic and other cantilever coatings, as well as for colloid probes. However, for several reasons, which depend on the specifics of the individual technique, measurement uncertainties for this approach tend to be at the high end of the range compared to other AFM calibration methods (currently estimated to be ± 10 to 30 %)[9].

In this work, we present a new method for measuring AFM cantilever probe spring constants using a microfabricated device called the Calibrated Reference Cantilever Array (CRCA).

2 MATERIALS AND METHODS

The Calibrated Reference Cantilever Array (CRCA), shown in Figure 1, was microfabricated from a Si (100) silicon-on-insulator (SOI) wafer[12]. The following describes the CRCA and its use to determine the spring constant of a commercial AFM Test cantilever of unknown stiffness.

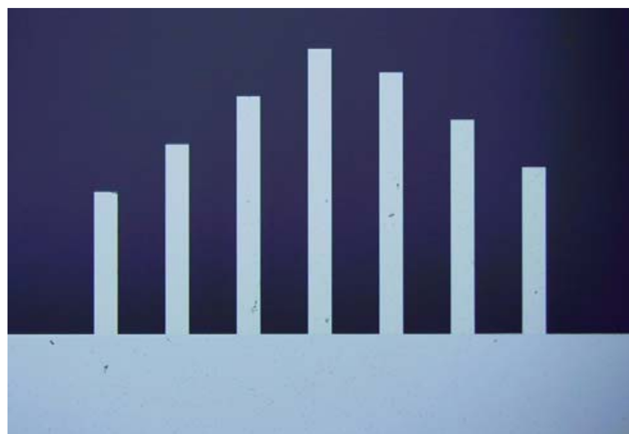

Figure 1: Scanning electron micrograph of the calibrated reference cantilever array (CRCA) manufactured from a Si (100) silicon-on-insulator (SOI) wafer. The cantilevers are nominally 1.4 μm thick, 50 μm wide and vary in length from 300 μm to 600 μm.

2.1 Reference Cantilever Array

The CRCA (Figure 1) was designed and microfabricated to minimize uncertainties in dimensional and material properties[12]. The arrays were created from single crystal Si (100) silicon-on-insulator (SOI) wafers using a series of deposition and etching processes combined with calibrated e-beam lithography to accurately align and pattern the cantilevers onto the SOI device layer of the wafer. Each array consists of seven rectangular cantilevers of identical width and thickness but different lengths (thus providing a different spring constant for each cantilever at its free end).

Table 1 shows normal spring constants for the seven reference cantilevers on the CRCA chip used for this work. Values were determined from material properties, as well as dimensional and resonance frequency measurements. The results are consistent with beam theory (k varies as L^{-3}) and are in good agreement with values obtained by independent measurements made using an electrostatic force balance device[12].

CRCA Cantilever Property	
***Length,* L (μm)**	***Normal stiffness,* k_{ref} (N/m)**
300	0.203
350	0.127
400	0.086
450	0.060
500	0.044
550	0.033
600	0.025

Table 1: Nominal spring constant values for CRCA cantilevers

2.2 Test cantilever and AFM instrument

All experimentation was carried out on a Digital Instruments Nanoscope IIIa AFM[13,14]. The "Test" cantilever used was a Veeco DNP type triangular cantilever ("D" = long, thin-legged)[13,14].

The normal spring constant of the DNP cantilever was independently calibrated via a '5 sphere' Cleveland added mass method (described in section 1). That is, 5 gold spheres[15, 14] of different size were placed onto and removed from the cantilever. The resonance frequency shift per added mass gave a k_{test} of (0.079 ±0.005) N/m[16,14].

2.3 Calibration procedure and results

The basic principle for calibrating the normal spring constant of an unknown "Test" cantilever using a "Reference" cantilever is depicted in Figure 2. In this work, the CRCA chip is attached to the AFM piezoelectric displacement transducer (henceforth called the "piezo"). For a known displacement of the piezo, the calibration is performed by recording two deflection measurements on the Test cantilever. One measurement provides δ_{piezo}, where the Test is in contact with an ideally stiff surface (in this case, the smooth bulk Si surface of the CRCA substrate). For this measurement, cantilever deflection tracks the piezo displacement. The other measurement provides δ_{cant}, where the Test is contacting a measured location (d_{load}) on a Reference cantilever (as depicted in Figure 2).

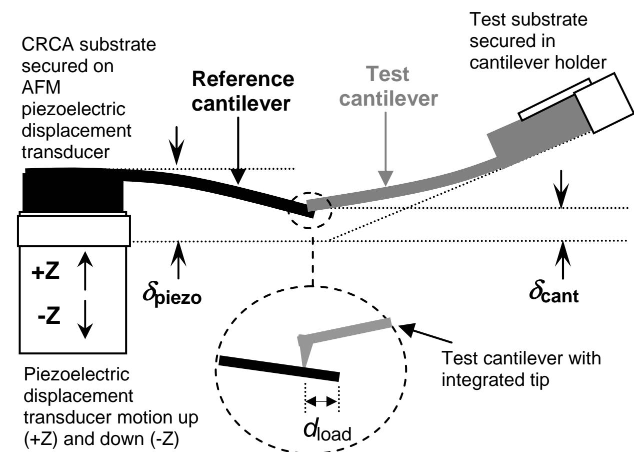

Figure 2: Schematic diagram for measuring the spring constant of an unknown Test cantilever using a calibrated Reference cantilever.

For an optical lever type AFM (as used here), the first measurement determines the output of the photodiode detector (proportional to cantilever deflection) as a function of Z (piezo) motion. This provides a sensitivity measurement for the optical lever system, which is then used to determine the deflection of the Test cantilever on the Reference cantilever in the second measurement. Data output appears as a "force-displacement curve". The 'approach' portion of a force-displacement curve is shown in Figure 3, consisting of an approach region where the surfaces remain out of contact, followed by a "compliance region" (upon contact). Referring to the compliance region, when a Test cantilever is in contact with an ideally rigid surface, and all deformation in the system occurs elastically in the cantilever, cantilever deflection and piezo (Z) displacement are equal, and a sensitivity for the optical lever system is obtained (S_{OL}, V/nm). When the Test cantilever is in contact with a compliant material (a Reference cantilever in this case), cantilever deflection and

piezo (Z) displacement are proportional, and a sensitivity for the Test—Reference Cantilever couple is obtained (S_{RC}, V/nm). In the experimental setup used for this work, the Test cantilever is mounted at an angle relative to the horizontal axis defined by the Reference cantilever (*ca.*11°; see Figure 2). The purpose of the current paper is to demonstrate precision in the calibration technique. As this mounting angle remained the same for all measurements in this work, the implications of this geometry are not addressed here. However, also referring to Figure 2, a factor that does affect measurement precision is the point of contact between the cantilevers, d_{load}. This geometrical parameter was measured and taken into account by the inclusion of an "off-end loading" correction[10].

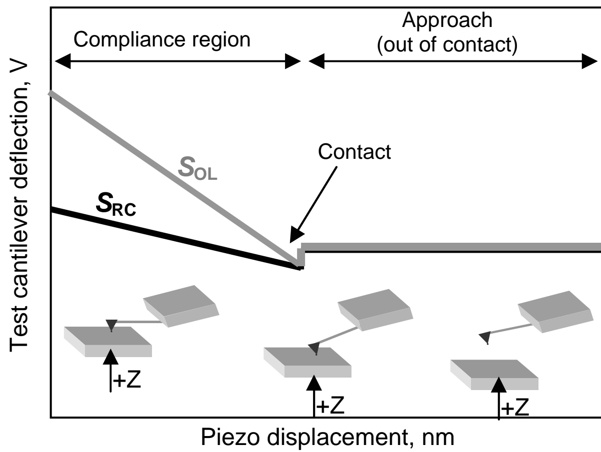

Figure 3: Force-displacement curve (approach only) examples for a Test cantilever contacting a rigid surface (S_{OL}) and a Reference cantilever (S_{RC}).

An array of seven reference cantilevers provides an opportunity to estimate the spring constant of the test cantilever in a more precise way. Correcting for off-end loading of a Reference cantilever, we can obtain the Test cantilever spring constant, k_{test}, through Hooke's law, such that

$$\alpha = \frac{k_{ref}}{k_{test}} = \left(\frac{S_{RC}}{S_{OL} - S_{RC}} \right) \left(\frac{L - d_{load}}{L} \right)^3 \tag{1}$$

A plot of α vs k_{ref} should give a straight line of slope $1/k_{test}$.

Using the commercial DNP "D" Test cantilever (described in 2.2), a series of 8 (repeat) S_{OL}–S_{RC} measurement pairs were conducted on each Reference cantilever. Figure 4 shows a plot of all data according to Equation 1. The slope of the linear regression best fit gives k_{test} = 0.080 N/m. Statistical analysis of the linear regression provides a standard uncertainty (standard error) of the slope of *ca* ±3 %[16,14]. This method of plotting the data according to Equation 1 is here called an 'array calibration'. On the other hand, the usual procedure for a reference artifact calibration is to press the AFM cantilever probe onto a single artifact to obtain a spring constant (called here a 'single-artifact calibration'). That is, for the same data as discussed above, we obtain k_{test} by treating each individual series of 8 (repeat) S_{OL}–S_{RC} measurement pairs on a single cantilever as an individual calibration (arranging Equation 1 such that $k_{test} = k_{ref}/\alpha$). For exactly the same data as shown in Figure 4, the single-artifact calibrations for all CRCA cantilevers yielded Test cantilever spring constants ranging from (0.073 to 0.086) N/m. The average value across this range was 0.081 N/m, with a 10 % standard deviation. The largest uncertainty observed for the shorter cantilevers (which can be seen from the data spread in Figure 4), is attributed to relative placement uncertainty (d_{load}), which contributes (to the third power) geometrically to the error.

The uncertainty in the determination of k_{test} using the array calibration method (±3 %) is a substantial improvement over both the single-artifact calibration method (±10 %) and the (five sphere) Cleveland added mass method (±6 %).

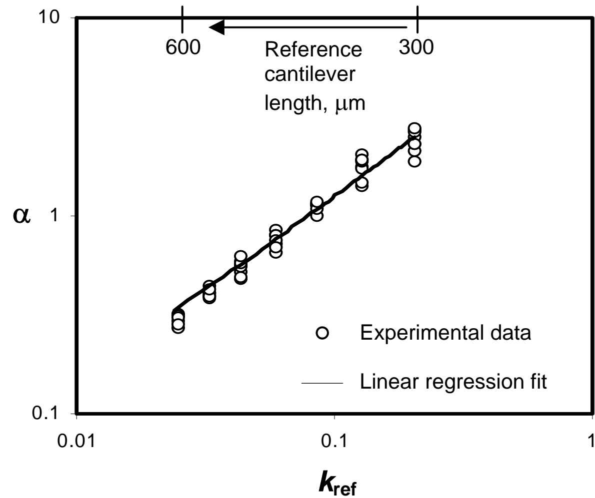

Figure 4: Reference cantilever array plot for a DNP-D test cantilever.

3 CONCLUSIONS

A new high precision technique for calibrating Atomic Force Microscope (AFM) cantilevers has been demonstrated using a unique microfabricated device called the Calibrated Reference Cantilever Array (CRCA). The calibration procedure allows the spring constant of an unknown cantilever to be obtained with greater precision compared with repeated measurements on a single cantilever. Substantial improvement in measurement uncertainty was also found over a popular dynamic response calibration method. The SI traceability potential of the device will further offer a high accuracy calibration method.

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[14] Certain commercial equipment, instruments, or materials are identified in this paper to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

[15] Alpha Aesar; purity 99.9 %.

[16] Slope of linear regression best fit and standard error of the slope were generated using Igor Pro (Wavemetrics, Inc).