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Variations in properties of atomic force microscope cantilevers fashioned from the same wafer

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Abstract
Variations in the mechanical properties of nominally identical V-shaped atomic force microscope (AFM) cantilevers sourced from the same silicon nitride wafer have been quantified by measuring the spring constants, resonant frequencies and quality factors of 101 specimens as received from the manufacturer using the thermal spectrum method of Hutter and Bechhoefer. The addition of thin gold coatings always lowers the resonant frequency but the corresponding spring constant can either increase or decrease as a result. The observed broad spread of spring constant values and the lack of correlations between the resonant frequency and spring constant can be attributed in part to the non-uniformity of composition and material properties in the thinnest dimension of such cantilevers which arise from the manufacturing process. The effects of coatings are dictated by the competing influence of differences in mass density and Young's modulus between the silicon nitride and the gold coating. An implication of this study is that cantilever calibration methods based on the assumption of uniformity of material properties of the cantilever in the thinnest dimension are unlikely to be applicable for such cantilevers.

S Supplementary data are available from stacks.iop.org/Nano/19/105709

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Microelectromechanical (MEM) devices often share the general attribute of having a quasi-two-dimensional form factor because one dimension is thin compared to the other two. When such devices are fashioned from the same wafer, there may be an expectation that the device properties are generally only dependent on the two planar dimensions. One of the most widely deployed example of such MEM devices is the micro-cantilever used in the atomic force microscope (AFM) for direct force measurements. Furthermore, gold coatings are applied to these cantilevers to enhance the sensitivity of optical methods used in measuring cantilever deflection. A key element in this application is the determination of the effective spring constant of the cantilever that is necessary for the conversion of measured deflections to physical forces. There are several cantilever geometries that are commonly employed and a number of different experimental approaches have been developed to determine cantilever spring constants [1–6]. Of these, the thermal method [1] has been incorporated in the system software by AFM manufacturers and is generally regarded as an industry standard method of calibrating cantilever spring constants because of its universal applicability to any cantilever geometry. In the thermal method [1, 7–9], the thermal power spectral density (PSD) function, $P(x)$, of the cantilever is measured as a function of circular frequency x. The resonant frequency, $f \equiv \omega/(2\pi)$, the quality factor, Q and the spring constant, K, of the cantilever is obtained by fitting the PSD function, $P(x)$, to the Lorentzian form corresponding to a simple harmonic oscillator. This method of determining the spring constant K is accurate provided the quality factor, Q, is much larger than unity, which is a condition that is generally well satisfied for measurements undertaken in air at atmospheric pressure.

It is plausible to assume that the properties of cantilevers fashioned from the same wafer would be similar although there have been no systematic statistical investigations of cantilever properties to substantiate this view. Existing studies have indicated variability of at least 20% in cantilever spring constants of the same type over ten to twenty cantilever samples [6, 10]. In addition Senden and Ducker [6] found that cantilever spring constants for commercial V-shaped silicon nitride cantilevers of the type studied in this work can differ by a factor of 4 between specimens taken from different wafers and Drummond and Senden [11] have shown that the Young's modulus of the silicon nitride films can differ by a factor of 2 between wafers as well.

Given the pivotal role of the cantilever spring constants in force measurements[6], we report measurements the spring constants, resonant frequencies, quality factors and physical dimensions of 101 specimens of commercially available V-shaped silicon nitride cantilevers using the thermal method and direct microscopic observations. All cantilevers are sourced from the same wafer. This is a sufficiently large sample size to obtain statistically significant results to characterize properties of nominally identical cantilevers. The effects of the application of additional thin gold coatings on cantilever properties will also be examined. Possible explanations on the variability of cantilever properties with and without additional coatings are proposed.

2. Experimental method

An Asylum MFP-3D atomic force microscope (Asylum Research, Santa Barbara, CA) was used for the collection of thermal power spectra of V-shaped silicon nitride cantilevers (NP-OHW, wafer no. 033803/22 Veeco Probes, CA[7]). All cantilevers were cleaned using UV/ozone for at least 15 min prior to measurements. The deflection sensitivity of each cantilever was averaged from at least five separate interactions with a solid glass substrate and this sensitivity was scaled by 1.09 [7, 8]. The power spectral density function was recorded using this sensitivity factor and fitted to a damped harmonic oscillator model with an allowance for white noise. The value of quality factor, Q, and resonant frequency, f, were determined and the spring constant calculated by the MFP-3D software (Igor Pro Version 5.05A, MFP3D version 050811 + 610) using the thermal tuning method, based on the theory of Hutter and Bechhoefer [1] There is no significant dependence in the calculated spring constant on the position of the cantilever in the holder. The physical dimensions of the cantilevers were measured using a Leica optical microscope (Leica Microsystems GmbH, Germany). An Emitech K575X (Emitech, UK) sputter coater was then used to coat the front side of a number of cantilevers first with chromium (~2 nm thick achieved at a current setting of 15 mA for 50 s) and then with gold (~10 nm thick achieved at a current setting of 15 mA for 50 s) to study the effects of the additional coating on cantilever properties.

Table 1. Summary of (a) mechanical properties and (b) physical dimensions (see figure 1) determined by the thermal method and optical microscopy of 101 V-shaped cantilevers derived from the same wafer as received from the manufacturer. Properties of individual specimens are available from the supplementary data (stacks.iop.org/Nano/19/105709).

(a)	Resonant frequency f (kHz)	Quality factor Q	Spring constant K (N m^{-1})
Mean	18.4	30.3	0.068
SD/mean	3.2%	5.2%	12%
Minimum	16.4	23.2	0.048
Maximum	20.1	33.2	0.11
(b)	Length L ($\times 10^{-6}$ m)	Base width b ($\times 10^{-6}$ m)	Leg width d ($\times 10^{-6}$ m)
Mean	193.3	202.8	23.3
SD/mean	0.32%	0.37%	2.6%
Minimum	191.2	201.1	22.3
Maximum	195.0	205.1	25.9

3. Results and discussion

A summary of the measured physical properties of 101 V-shaped cantilevers, used as received from the manufacturer without further coating, is given in table 1. Values pertaining to individual samples are available from the supplementary data (stacks.iop.org/Nano/19/105709). The most striking observations is that, while the planar dimensions of the sample population are very uniform with a small standard deviation of less than 1% relative to the mean in the larger dimensions, the spring constant varies by more than a factor of 2 between the sample minimum and maximum values, with a standard deviation of 12% of the mean.

The actual distribution of spring constant, K, values is shown in figure 1. The minimum and maximum value of the 101 specimens ranged from 0.048 N m^{-1} to 0.11 N m^{-1}. Approximately one-third (34/101) of the specimens have spring constants between the range 0.065–0.070 N m^{-1} with a similar proportion with spring constants below (31/101) and above (36/101) this range, and about 70% or (73/101) fall in the middle band, 0.060–0.075 N m^{-1}. According to manufacturer specifications, the nominal value of the spring constant in this batch is 0.06 N m^{-1}. It is worth noting that

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[6] A private communication from J E Sader, P Mulvaney and W A Ducker noted the possibility of an anomalous relation between the spring constant and resonant frequency of the cantilever used in a recent AFM measurement of dynamic forces between emulsion drops in an aqueous electrolyte [19].

[7] The manufacturer's specifications are thickness: 0.4–0.7 × 10^{-6} m, resonant frequency: 12–24 kHz, spring constant 0.06 N m^{-1}, backside coating: Cr/Au 15 nm/60 nm, front side: uncoated.

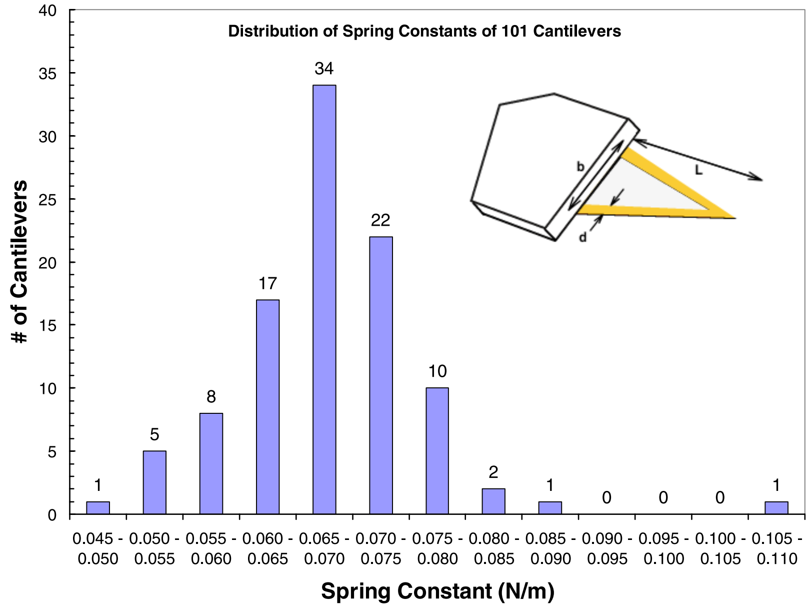

Figure 1. Distribution of spring constants as measured by the thermal method of 101 V-shaped cantilevers fashioned from the same wafer as received from the manufacturer.

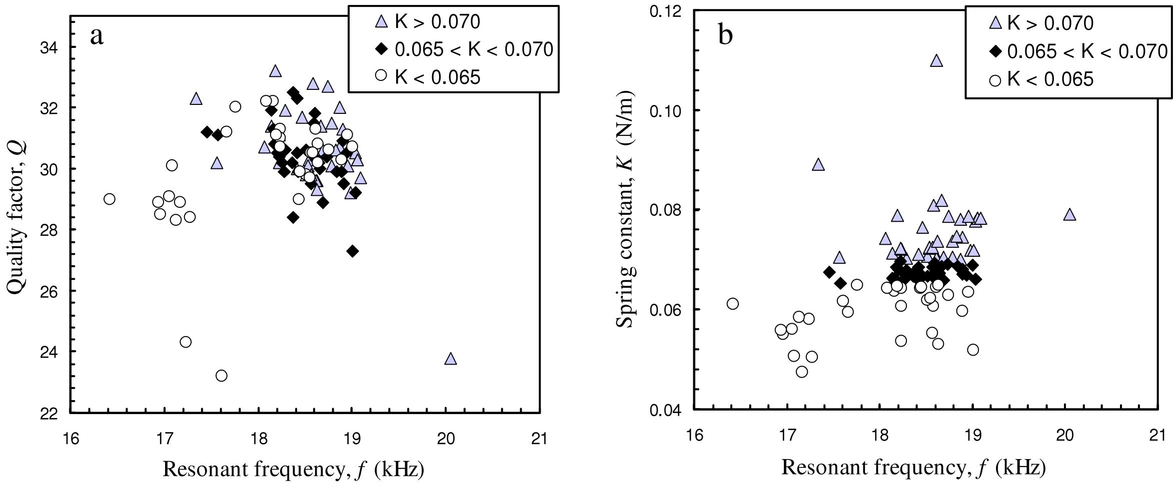

Figure 2. (a) Variation of measured quality factor, Q, with resonant frequency, f (kHz), for specimens with spring constants, $K < 0.065$ N m^{-1} (○), $0.065 < K < 0.070$ N m^{-1} (◆) and $K > 0.070$ N m^{-1} (▲) among 101 as-received V-shaped cantilevers fashioned from the same wafer as determined by the thermal method. (b) Variation of measured spring constant, K (N m^{-1}), with resonant frequency, f (kHz), for the same specimens with the same meaning of the symbols.

for all the cantilevers measured, the resonance frequencies are within the range specified by the manufacturer, but the manufacturer only reports a nominal value for the spring constant and not a range of values. The implication is that, if quality control on these cantilevers is measured by resonance frequency, the manufacturer has met the product specifications even with a larger variation in spring constant values.

From figure 2, we see that the quality factor, Q, and the spring constant, K, are not well correlated to the resonant frequency, f, although the data may suggest a weak tendency that cantilevers with higher resonant frequencies may perhaps have higher spring constants. This lack of a close correlation between spring constant and resonant frequency is consistent with finite element modelling predictions of spring constants for V-shaped cantilevers with gold coatings [12] where it is observed that the spring constant is expected to increase monotonically as a function of resonant frequency with an increase in the thickness of the silicon nitride layer, but the resonant frequency shows an inverse relation with spring constant with an increase in the gold coating thickness. As received from the manufacturer, the cantilevers have a nominal coating of Cr/Au (15 nm/60 nm). Therefore variation in the thickness of either the silicon nitride or gold coating results in competing effects on the behaviour of the spring constant with resonance frequency leading to the scatter observed in figure 2(b).

To demonstrate the effects of the coating on resonant frequency, the results of taking 17 cantilevers of the original

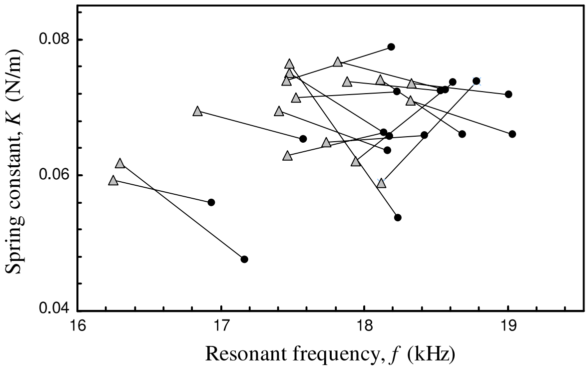

Figure 3. Variation of measured spring constant, K (N m^{-1}), with resonant frequency, f (kHz), for specimens with spring constants before and after the addition of Cr/Au coatings (2 nm/10 nm) on the front surface of the cantilever. The uncoated values (●) are connected to the corresponding coated value (▲) for each cantilever.

101 sample and adding a Cr/Au (2 nm/10 nm) coating to the front surface of the cantilever using a sputter coater are given in figure 3. In all cases the resonant frequency decreases with the additional coating, but the spring constant in each case either increased or decreased or remained unchanged. It is unlikely that the spring constant of a cantilever would decrease with the addition of a metal coating. The change in spring constant upon coating is small in most cases, well within the error of the measurement, indicating that the thin layer of gold has little or no effect on the spring constant. As discussed in the finite element study by Hazel and Tsurkruk [12] the Young's modulus of silicon nitride is much higher than that of gold, whereas the density of silicon nitride is reported to be 3000 kg m^{-3} and gold is 19 600 kg m^{-3} [12]. Thus the addition of gold increases the mass significantly and therefore depresses the resonant frequency while having a smaller effect on the spring constant. It is also possible that interfacial stresses from the addition of the coating may cause the cantilever to bend and affect the cantilever spring constant, but this is expected to become significant for thicker coatings [13].

The processing of the silicon nitride cantilever may also contribute to the variability in the material properties of these cantilevers. The manufacturing process of this type of cantilever is through low-pressure chemical vapour deposition (LPCVD) [14, 15]. The growth of a silicon nitride film on a silicon substrate can often leave residual stresses in the film, from both intrinsic stress from the microstructure in the film and from the thermal expansion mismatch between the silicon nitride film and the silicon substrate as LPCVD operates at temperatures as high as 800 °C [14, 16]. These stresses are commonly minimized in the manufacture of micromechanical devices and cantilevers, but residual stresses can remain in the material [17]. The silicon nitride cantilever is also bonded to a glass substrate. The glass substrate is bonded to the silicon nitride pattern so that the edge of the glass substrate is aligned with the base of the cantilever. Variability in the alignment of the glass edge can result in a thin region of silicon nitride film protruding beyond the edge of the glass at the base or root of the cantilever. This is commonly visible on most commercial cantilevers as well as in the original paper that discussed their manufacture [14]. Recent studies on silicon nitride rectangular cantilevers manufactured without the glass block, but with the same type of structure at the root, have shown this region can be compliant and can affect the cantilever stiffness [18]. The thermal method is not limited to a particular geometry or an ideal root condition. The effective stiffness for the cantilever is measured where the compliance of the root may contribute to the overall compliance of the cantilever. If the thickness of the compliant region at the root of the cantilever is significant in comparison to the length of the cantilever [18] this may have a small systematic effect on the scaling factor used on the detector sensitivity in the thermal method [7, 8]. The root condition may become a much larger difficulty in models that assume an ideal root condition for the cantilever [18].

The applicability of a calibration method is dependent on any assumptions made about the physical properties of the cantilever. If the material properties of the cantilever fail to meet these assumptions, for example in anisotropic material properties or non-ideal root condition, then the use of that calibration method may not be appropriate. A recent heuristic scaling method has been proposed [13] where it was suggested that the spring constants of any arbitrary shape cantilevers can be calculated accurately from measured values of the resonant frequency and quality factor once a characteristic hydrodynamic function of the cantilever is known. This heuristic approach is an extension of the well-known 'Sader method' [5] for cantilever calibration, developed for rectangular cantilevers, to other cantilever geometries, namely the triangular or V-shaped cantilever. A key requirement of this method is that the hydrodynamic function of the cantilever only depends on the 2D planar shape of the cantilever, but not on the absolute dimensions, so that this function is universal for all cantilevers of the same shape and aspect ratio. The method therefore assumes an ideal root condition for the cantilever and that the material properties of the cantilever are isotropic and uniform in the thinnest dimension. The theoretical foundation of this heuristic approach for arbitrary geometries appears sound but the method has not been fully tested for commercial V-shaped cantilever with gold coatings.

The basis of the scaling method proposed by Sader *et al* [13] for calibrating AFM cantilevers can be summarized by the following scaling relation for the spring constant, K:

$$K = \rho d^2 L \omega^2 Q \Omega(Re) \tag{1}$$

which depends on the planar dimensions length, L, and the leg width, d, of the V-shaped cantilever. Because the wafer thickness, h, is small compared to all the other dimensions of the cantilever the properties in the thinnest dimension are assumed to be uniform so that K does not depend explicitly on the cantilever wafer thickness. The planar shape of the cantilever is encoded in the hydrodynamic function $\Omega(Re)$ which is assumed to be a function of the Reynolds number $Re = (\rho d^2 \omega/\eta)$, where ρ is the mass density (1.18 kg m^{-3}) and η is the viscosity (1.86×10^{-5} Pa s) of air at the atmospheric conditions under which the present experiment is carried out [13].

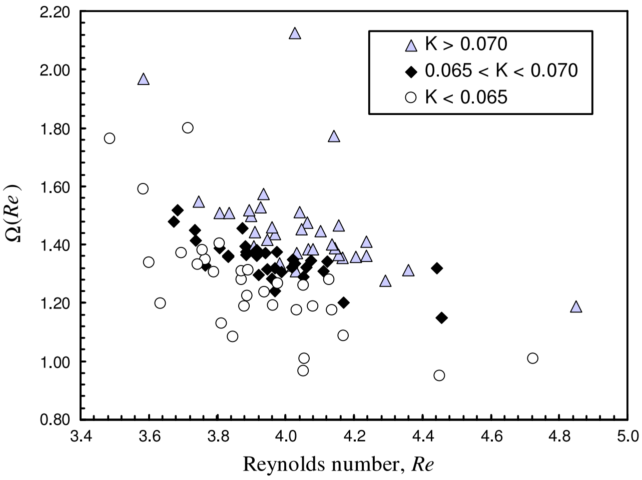

Figure 4. Variation of the hydrodynamic function $\Omega(Re)$ with Reynolds number Re for specimens with spring constants, $K < 0.065$ N m^{-1} (○), $0.065 < K < 0.070$ N m^{-1} (◆) and $K > 0.070$ N m^{-1} (▲) among 101 as-received V-shaped cantilevers fashioned from the same wafer as determined from equation (1) and physical parameters obtained by the thermal method and direct microscopic measurement.

Equation (1), in particular the function $\Omega(Re)$, has been measured over almost three decades of the Reynolds number at 8–10 different values by varying the gas pressure. The results are then fitted to an analytical function on a $\log(Re)$ scale [13] This has been carried out for two cantilever specimens of different shapes. The resulting $\Omega(Re)$ functions are then used to predict spring constants of cantilevers of the same shape using equation (1) and measured values of the resonant frequency and quality factor. It is worth noting that, although the hydrodynamic function $\Omega(Re)$ was measured over three decades of the Reynolds number, Re, the measured values of the resonant frequencies and quality factors used to predict the spring constant were taken over a range of the Reynolds number of the order of 1 which is only subtended by two calibration data points for $\Omega(Re)$.

Since our present measurements have been taken over a statistically significant sample size we have sufficient data to study the form of the hydrodynamic function $\Omega(Re)$ which, if the scaling theory is applicable, should yield $\Omega(Re)$ as a smooth function of the Reynolds number, Re. In figure 4, we show the function $\Omega(Re)$ for the 101 specimens of V-shaped cantilevers over the range $3.4 < Re < 5.0$ under which our measurements were conducted. The range of Reynolds numbers (reported in the supplementary data (available at stacks.iop.org/Nano/19/105709)) was dominated by the changes in resonance frequency as planar dimensions varied little over the cantilever dataset. The hydrodynamic function $\Omega(Re)$ is calculated from equation (1) as all other parameters in the equations can be measured. From the result in figure 4 there appears to be no simple correlation between Ω and Re although for a given value of the Reynolds number, Re, cantilevers with higher spring constants tend to have a higher value of Ω.

This scaling method is susceptible to any of the variability issues discussed above. The difficulties in applying this type of scaling method to silicon nitride cantilevers have been observed previously for rectangular cantilevers. Two very thorough studies comparing cantilever calibration methods for rectangular cantilevers have found the Sader method to deviate from both the added mass method (or Cleveland method) as well as thermal methods by as much as 70% for silicon nitride cantilevers with gold coatings [3, 10]. Burnham and coworkers [3, 10] had attributed this deviation to the effects of the gold coating, but the processing issues discussed above may contribute as well.

4. Conclusions

In this paper we have undertaken a statistically significant study of the correlation between mechanical properties such as resonant frequency, quality factor and spring constant of V-shaped cantilevers which is crucial for AFM force measurement applications. For silicon nitride cantilevers fashioned from the same wafer, we found variations of spring constant values over a factor of two even though the geometric dimensions of all specimens are almost identical. Furthermore there is no systematic correlation between the resonant frequency and the spring constant. Moreover, the application of additional gold coatings to the cantilever will decrease the resonant frequency but can either increase or decrease the spring constant. The variability of the spring constants and hydrodynamic functions of these cantilevers is attributed to the competing effects between the higher density of the gold coating and the higher Young's modulus of the silicon nitride as well as the manufacturing process for silicon nitride cantilevers. The results of these measurements facilitated the quantification of the scaling method proposed to calibrate cantilevers. The lack of a well-defined correlation between the hydrodynamic function, $\Omega(Re)$, and the Reynolds number, Re, which is central to the applicability of the scaling method, suggests that scaling methods should be used with caution for this family of cantilevers. The deviations in $\Omega(Re)$ are consistent with the observation of deviations of the Sader method from the thermal method for rectangular silicon nitride cantilevers with gold coatings [3, 10]. The studies by Burnham *et al* [3, 10] and the present work do not detract from the validity of the theory developed by Sader; these studies simply suggest caution when using the Sader methods for cantilevers made with silicon nitride and gold coatings.

Acknowledgments

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References

[1] Hutter J L and Bechhoefer J 1993 *Rev. Sci. Instrum.* **64** 1868
[2] Cleveland J P, Manne S, Bocek D and Hansma P K 1993 *Rev. Sci. Instrum.* **64** 403

[3] Burnham N A, Chen X, Hodges C S, Matei G A, Thoreson E J, Roberts C J, Davies M C and Tendler S J B 2003 *Nanotechnology* **14** 1

[4] Torii A, Sasaki M, Hane K and Okuma S 1996 *Meas. Sci. Technol.* **7** 179

[5] Sader J E, Chon J W M and Mulvaney P 1999 *Rev. Sci. Instrum.* **70** 3967

[6] Senden T J and Ducker W A 1994 *Langmuir* **10** 1003

[7] Jaschke M and Butt H J 1995 *Nanotechnology* **6** 1

[8] Walters D A, Cleveland J P, Thomson N H, Hansma P K, Wendman M A, Gurley G and Elings V 1996 *Rev. Sci. Instrum.* **67** 3583

[9] Hutter J L, Chen J, Wan W K, Uniyal S, Leabu M and Chan B M C 2005 *J. Microsc.* **219** 61

[10] Matei G A, Thoreson E J, Pratt J R, Newell D B and Burnham N A 2006 *Rev. Sci. Instrum.* **77** 083703

[11] Drummond C J and Senden T J 1995 *Mater. Sci. Forum* **189** 107

[12] Hazel J L and Tsukruk V V 1999 *Thin Solid Films* **339** 249

[13] Sader J E, Pacifico J, Green C P and Mulvaney P M 2005 *J. Appl. Phys.* **97** 124903

[14] Albrecht T R, Akamine S, Carver T E and Quate C F 1990 *J. Vac. Sci. Technol.* A **8** 3386

[15] Khan A, Philip J and Hess P 2004 *J. Appl. Phys.* **95** 1667

[16] Resnik D, Aljancic U, Vrtacnik D, Mozek M and Amon S 2005 *Vacuum* **80** 236

[17] Martyniuk M, Antoszewski J, Musca C A, Dell J M and Faraone L 2006 *Smart Mater. Struct.* **15** S29

[18] McShane G J, Boutchich M, Phani A S, Moore D F and Lu T J 2006 *J. Micromech. Microeng.* **16** 1926

[19] Dagastine R R, Manica R, Carnie S L, Chan D Y C, Stevens G W and Grieser F 2006 *Science* **313** 210