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Temperature and damping effects on the frequency dependence of electrostatic force microscopy force gradients

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In this paper we demonstrate that depending on the surrounding ambient, which may be manifested by different damping conditions, a “bump” in frequency response of Electric Force Microscope (EFM) cantilevers may be observed, either in electrical amplitude or phase signals of AC gradients. This bump is present in air and at room temperature when the cantilever oscillates at fundamental frequency with high excitation amplitude but disappears at increasing temperature. This effect can be explained by a significant temperature decrease of the Q factor of coated cantilevers. At a constant value of AC voltage, the relevant parameter is the ratio of Q factor by cantilever stiffness (of the nth oscillation mode) from which we can predict bump observation. This study highlights significant effects for quantitative EFM studies under different atmospheres and at different temperatures. © 2013 AIP Publishing LLC. [http://dx.doi.org/10.1063/1.4843835]

I. INTRODUCTION

Since its emergence in the earlier 1990s,1 electrostatic force microscopy (EFM) is in constant development, whether in force2 or gradient3 detection mode, both in air or vacuum4 environment. A recent milestone was reached with the use of high flexure modes of vibration for amplitude-controlled atomic force microscopy AFM (AM-AFM or TappingTM) under vacuum.5 The stabilization time threshold of the oscillating sensor was identified as a key point that can be reduced by increasing the mode order. It was shown experimentally that, in both air and vacuum, the stabilization time decreases notably when the order of the flexure mode of the cantilever increases. Under ambient conditions, the result is a possible improvement of scan speeds by about one order of magnitude. Under vacuum and using standard sensors, the amplitude-controlled conditions are satisfied for resonance modes equal to or higher than the second. The feasibility of detecting electrostatic gradients in the linear regime has then been shown under vacuum by AM-AFM and a double pass method.6 To achieve this goal, different flexure mode orders were employed. It was shown that the sensitivity of the frequency or phase shifts to a given gradient was reduced when the order was increased. Gradient detection, on the basis of different flexure mode orders, was thus extended from controlled atmosphere to vacuum.

Another milestone was reached by Crider et al.7,8 and Riedel et al.9 who were the first to develop the so-called Local Dielectric Spectroscopy (LDS). The technique, based on the detection of the phase shifts of AC gradients, was developed using FM-AFM under vacuum and was successfully applied to the study of dielectric losses (tan delta) in thin polymer layers. Another approach, called nanoDielectric Spectroscopy, has recently been adapted by Schwartz et al.10 in ambient conditions, by detecting AC forces in the standard AM-AFM mode over 4 decades.

Despite such advances, EFM signals require a rigorous analysis. Namely, this means that it is not possible to apply any signal with any amplitude at any frequency without having an impact on the interpretation of the images. In a previous work, AC force gradients detected under vacuum have been shown to exhibit a first order filter behaviour.11 The conclusion was that to get acceptable signal magnitudes, a good compromise must be obtained between the choices of the mode order and the electrical frequency since due to $Q_0/k_0$ ratio (where $Q_0$ and $k_0$ are, respectively, the quality factor and the cantilever stiffness of the nth oscillation mode, $n = 0$ corresponding to the fundamental mode), the signal is reduced when the mode order increases. In this work we demonstrate that depending on the surrounding ambient, which may be manifested by different damping conditions, a “bump” in frequency response may also be observed, either in electrical amplitude or in phase signal. This bump is present at room temperature when the cantilever oscillates at fundamental frequency with high electrical amplitude but disappears at increasing temperature. This effect can be explained by a significant temperature decrease of the Q factor of coated cantilevers (such as those used in EFM operations) with temperature.

Klempner et al.12 have shown, in particular, that the film thickness has a significant effect on the Q factor. They noticed that Q factors of uncoated microcantilevers was invariant with temperature while microcantilevers coated (Al 30 nm) on one side as well as those coated on both sides experienced substantial reduction in Q factor (A reduction of...
about 35% from 10°C to 90°C). Choi et al. have also shown that double-layered microcantilevers operating in deflection mode can reach extreme thermal sensitivities. Stress can be induced thermally in thin film multilayer structures due to the difference in the thermal expansion coefficients between the adjacent layers or that the structures are subjected to temperature changes during their manufacturing and subsequent use.

Very recently, Dunaevskiy et al. put into evidence another important thermal effect on bilayer cantilevers. They proposed a technique consisting in using the probe itself as a photodetector to visualize infrared radiation. The heat generated in the probe by absorbed laser radiation modifies mechanical properties of the cantilever, and this interaction is detected at low voltages. Different flexure modes and compare them to simulations. We then study the relationship between mechanical phase shifts and compare them to simulations. We then study the experimental phases and amplitudes and compare them to simulations. We then study the relationship between the appearance of a bump on the phase and force gradients, assuming 

\[ \Delta \phi_{\text{AC}} \approx \frac{\Delta \phi_0}{\sqrt{1 + (a \omega_n)^2}}, \]

and their cut frequencies, defined by \( f_{s_{\omega_n}} = \frac{1}{2\pi} \) and \( f_{s_{2\omega_n}} = \frac{1}{4\pi} \), differed by a factor of two \( \tau_n = \frac{Q_n}{f_n} \) corresponding to the stabilization time at the mode order \( n \). An example of cantilever excited under vacuum on the second mode (\( f_1 \approx 442 \text{ kHz} \)) was presented. The experimental quality factor was \( Q_1 \approx 19 \text{ kHz} \), and the dynamic stiffness was estimated (by using the formula \( k_n = (f_n/f_0)^2 k_0 \), with \( k_0 \approx 2 \text{ N/m} \) and \( f_0 \approx 70 \text{ kHz} \)) to be about \( k_1 \approx 80 \text{ N/m} \). The ratio \( Q_1/k_1 \approx 250 \) was of the same order of magnitude than those obtained using the fundamental mode in air (\( Q_0/k_0 \approx 150 \)). This helped to explain why the sensitivity using the second mode under vacuum was as good as the sensitivity obtained in air on the fundamental mode.

This work was a starting point, and yet it was not possible to generalize this observation to all experimental configurations potentially encountered and to take into account, for example, a possible change in the vibration mode or atmosphere. To better understand all these different situations we propose in what follows a complete spring-mass modelling of the cantilever dynamics in the presence of AC electrostatic force gradients.

C. Modelling

1. General case

In the spring-mass approximation and a linear oscillating system, where mechanical and electrical forces are brought into play, the tip position \( \delta z(t) \) over time is governed by the equation

\[ m_n \ddot{\delta} + a_n \dot{\delta} + \left[ k_n - (G_0 + G_1 \sin(\omega_n t) - G_2 \cos(2\omega_n t)) \right] \dot{\delta} = F_0 - k_n (d - z_0) + F_1 \sin(\omega_n t) - F_2 \cos(2\omega_n t) + F_M \sin(\omega_M t). \]

\( \omega_n \) and \( \omega_M \) are, respectively, the mechanical and electrical angular frequency, \( F_0, F_1 \) and \( F_2 G_0; G_1 \) and \( G_2 \) are the static, \( \omega_e \) and \( 2\omega_e \) frequency components of the electric force (and force gradient, respectively). \( k_n \) and \( z_n \) are, respectively, the dynamic stiffness and damping coefficient of the cantilever at the mode order \( n \). The general expression of \( \dot{\delta} z(t) \) can be expressed as

\[ \dot{\delta} z(t) = X(\omega_mt) \cos(\omega_mt) + Y(\omega Mt) \sin(\omega_M t), \]

where

\[ X(t) = X_0 + X_A \cos(\omega_et) + X_B \sin(\omega_et) + X_C \cos(2\omega_et) + X_D \sin(2\omega_et), \]

with morphology with an rms noise in the millivolt range. When an AC plus DC tip-to-sample voltage is applied, since the gradient is proportional to the square voltage, two ac phase components at \( \omega_e \) and \( 2\omega_e \) frequencies are expected to be observed. Experimental phases \( \Delta \phi_{\omega_e} \) and \( \Delta \phi_{2\omega_e} \) versus the electrical frequency were detected with a cantilever vibrating at the second flexure mode. For both cases, a first-order filter behaviour was clearly observed, according to the following expression

\[ \Delta \phi_{\omega_e} = \Delta \phi_0 / \sqrt{1 + (a \omega_e)^2}, \]

and the following expression

\[ \Delta \phi_{2\omega_e} = \Delta \phi_0 / \sqrt{1 + (a 2\omega_e)^2}. \]
By inserting these expressions into Eq. (1) and by identifying the terms with the same combinations of electrical and mechanical frequencies, the expressions of the different coefficients $X_0, X_A, X_B, \ldots, Y_0, Y_A, Y_B, \ldots$ can be obtained after solving independently three different systems (6)–(8) of equations.

\[
\begin{align*}
-\frac{1}{2} & Y_0 \omega^2 \frac{m^2}{a^2} - Y_0 \omega^2 \frac{m^2}{b^2} + X_0 \omega_x x_n - 2X_0 \omega_x \omega_m m_n + Y_0 \omega_x x_n + (k_n - G_0) Y_A = 0 \\
-\frac{1}{2} & Y_0 \omega^2 \frac{m^2}{a^2} - X_0 \omega_x x_n - 2X_0 \omega_x \omega_m m_n + Y_0 \omega_x x_n + (k_n - G_0) Y_B = 0 \\
2 & X_0 \omega_x \omega_m m_n - Y_0 \omega_x \omega_m m_n + 2Y_0 \omega_x \omega_m m_n + Y_0 \omega_x \omega_m m_n + (k_n - G_0) Y_B = 0 \\
-\frac{1}{2} & X_0 \omega^2 \frac{m^2}{a^2} - X_0 \omega_x x_n - 2X_0 \omega_x \omega_m m_n + Y_0 \omega_x x_n + (k_n - G_0) Y_B = 0 \\
-\frac{1}{2} & X_0 \omega^2 \frac{m^2}{a^2} - X_0 \omega_x x_n - 2X_0 \omega_x \omega_m m_n + Y_0 \omega_x x_n + (k_n - G_0) Y_B = 0 \\
& X_0 \omega_x \omega_m m_n - Y_0 \omega_x \omega_m m_n + 2Y_0 \omega_x \omega_m m_n + Y_0 \omega_x \omega_m m_n + (k_n - G_0) Y_B = 0.
\end{align*}
\]

System (7) (cos $\omega t \sin \omega_m t$, sin $\omega t \cos \omega_m t$, sin $\omega t \sin \omega_m t$, cos $\omega t \cos \omega_m t$ combinations) provides the solutions of $X_A, Y_A, X_B$, and $Y_B$.

\[
\begin{align*}
-4Y_C \omega^2 \frac{m^2}{a^2} - X_C \omega x_n - 2X_D \omega x_n - 4Y_C \omega x_n + Y_D \omega x_n + (k_n - G_0) Y_C + G_2 Y_0 = 0 \\
-4Y_D \omega^2 \frac{m^2}{a^2} - X_D \omega x_n - 2Y_C \omega x_n + Y_D \omega x_n + (k_n - G_0) Y_D = 0 \\
-4Y_D \omega^2 \frac{m^2}{a^2} - Y_D \omega x_n + 2X_C \omega x_n + 2Y_C \omega x_n + (k_n - G_0) Y_D = 0 \\
-4X_C \omega^2 \frac{m^2}{a^2} - X_C \omega x_n - 2X_D \omega x_n + 2Y_C \omega x_n + (k_n - G_0) Y_C = G_2 X_0 = 0.
\end{align*}
\]

System (8) (cos $2\omega t \sin \omega_m t$, sin $2\omega t \cos \omega_m t$, sin $2\omega t \sin \omega_m t$, cos $2\omega t \cos \omega_m t$) provides the solutions of $X_C, Y_C, X_D$, and $Y_D$.

The in-phase and out-of-phase components, $X(t)$ and $Y(t)$, can express, respectively, as

\[
\begin{align*}
X(t) &= X_0 + \sqrt{X_A^2 + X_B^2} \cos(\omega t - \arctan(X_B/X_A)) \\
&\quad + j\sqrt{X_A^2 + X_B^2} \sin(\omega t - \arctan(X_B/X_A)) \\
&\quad + \sqrt{X^2 + X_D^2} \cos(2\omega t - \arctan(X_D/X_C)) \\
&\quad + j\sqrt{X^2 + X_D^2} \sin(2\omega t - \arctan(X_D/X_C)),
\end{align*}
\]

\[
\begin{align*}
Y(t) &= Y_0 + \sqrt{Y_A^2 + Y_B^2} \cos(\omega t - \arctan(Y_B/Y_A)) \\
&\quad + j\sqrt{Y_A^2 + Y_B^2} \sin(\omega t - \arctan(Y_B/Y_A)) \\
&\quad + \sqrt{Y^2 + Y_D^2} \cos(2\omega t - \arctan(Y_D/Y_C)) \\
&\quad + j\sqrt{Y^2 + Y_D^2} \sin(2\omega t - \arctan(Y_D/Y_C)).
\end{align*}
\]

The origin of the phases, being taken at the resonance frequency, the mechanical phase shifts (proportional to the total electric force gradient) are such that $\tan \Delta \phi_m = -Y(t)/X(t)$. In the general case, $\tan \Delta \phi_m$ can be then decomposed automatically using Maple software into three complex amplitudes ($DC, \omega_c$, and $2\omega_c$)

\[
\tan \Delta \phi_m = \tan \Delta \phi_{DC} + \tan \Delta \phi_{\omega_c} + \tan \Delta \phi_{2\omega_c} = \tan \Delta \phi_{DC} + \text{amp}_{\omega_c} e^{\text{phase}_{\omega_c}} + \text{amp}_{2\omega_c} e^{\text{phase}_{2\omega_c}}.
\]
\( \omega_e \) and \( 2\omega_e \) cut frequencies can be then deduced

\[
 f_{c_{\omega_e}} = \frac{1}{2\pi} \text{ and } f_{c_{2\omega_e}} = \frac{1}{4\pi}.
\]

In the small angle approximation, \( \tan \Delta \phi_m \approx \Delta \phi_m \), valid for low AC gradients, relation (11) can thus explain the first order behaviours that where observed experimentally under vacuum using the second mode by Portes et al.\(^{11}\)

The relationship between the detected phase and gradient can be generalized under the form

\[
\Delta \phi_e \approx -\frac{Q_n}{k_n} g(f_e) G_i, \tag{16}
\]

where \( g(f_e) = 1 \) DC component and \( g(f_e) = \frac{1}{\sqrt{1+4f_i^2}} \) or \( \frac{1}{\sqrt{1+16f_i^2}} \) for \( \omega_e \) and \( 2\omega_e \) components, respectively.

III. OBSERVATIONS OF AC GRADIENTS IN AIR ATMOSPHERE AT DIFFERENT TEMPERATURES USING 1ST RESONANCE (FUNDAMENTAL MODE)

A. Set-up

The experimental set-up allowing detection of amplitude and phase of \( \omega_e \) and \( 2\omega_e \) components of AC gradients is described in Figure 1. The measurements are performed using AM-AFM with a cantilever vibrating at the mechanical angular frequency \( \omega_m \) close to its resonance frequency, in front of a metallic plate. The electrical excitation at the angular frequency \( \omega_e \) is superimposed to the mechanical one. According to relation (1), due to both AC and DC voltages, the mechanical phase shift \( \Delta \phi_m \) is introduced and is in principle available, in most commercial devices, at the output of the AFM controller via a signal access modulus. AC components of mechanical phase shifts (i.e., electrical amplitude and phase shift at \( \omega_e \) and \( 2\omega_e \), respectively) can be detected by means of a lock in amplifier with \( \omega_e \) as reference frequency. To better control the tip sample distance and to decorrelate the measurement from topography, AM-AFM was realized using the so-called “double pass method.” During the second pass the tip is retracted from a “lift height,” the mechanical amplitude is reduced in order to maintain the cantilever in a linear regime and the tip-sample distance is approximately equal to the first pass amplitude (determined from an amplitude distance curve) plus the lift height. AC and DC voltages are then switched-on and AC gradients demodulated. A duration of the second pass of several seconds is required at low electrical frequencies in order to be adapted with time constants of the lock in amplifier which must be equal to at least 2 or 3 periods.

B. Low gradients (\( V_{AC} = 1 \text{ V} \)) simulations: Amplitude and phase signals (\( \omega_e \) and \( 2\omega_e \) components)—Comparison with literature

We first investigated low gradients effects on AC phase shifts and amplitude signals. Low AC voltages around \( V_{AC} = 1 \text{ V} \) were considered. For such voltages, with a tip-sample distance \( z = 25 \text{ nm} \), a tip radius \( R = 50 \text{ nm} \), a cone angle of about 15° and a DC voltage of 0.5 V, typical values of gradient components were found (according to Fumagalli’s model\(^{15}\)) to be about \( G_0 \approx 3 \times 10^{-3} \text{ N/m} \), \( G_1 \approx 2 \times 10^{-3} \text{ N/m} \), and \( G_2 \approx 1 \times 10^{-3} \text{ N/m} \). Simulations performed on the fundamental mode (\( n = 0 \)) in air with standard cantilever (\( f_0 = 70 \text{ kHz}, k_0 = 2 \text{ N/m}, Q_0 = 250 \)) are presented in Figure 2. These parameters are the same than those described by Portes et al.\(^{11}\) A filter-like behavior of the first order is characterized through \( \omega_e \) and \( 2\omega_e \) amplitudes (Figure 2(b)) with cutoff frequency values of about \( f_{c_{\omega_e}} = 111 \text{ Hz} \) et \( f_{c_{2\omega_e}} = 61 \text{ Hz} \) and phase shifts of 45° (with respect to low frequencies) at these frequencies.

The results obtained by simulation of the fundamental mode amplitude yield comparable to those obtained experimentally under vacuum using the second mode (\( n = 1 \)) results (\( f_1 = 442 \text{ kHz}, k_1 = 80 \text{ N/m}, Q_1 = 20000 \)).\(^{11}\) In both cases \( Q_0/k_0 \) are of the same order of magnitude (\( \approx 150–250 \)); the magnitude of this ratio could be thus responsible for such an effect and will be under study in Secs. III C and IV. The criterion of weak or strong gradient is not yet clearly established and will be the subject of further discussions in Secs. III C and IV.

C. High gradients (\( V_{AC} = 5 \text{ V} \)): Amplitude and phase signals (\( \omega_e \) and \( 2\omega_e \) components)—Comparison with modelling

To study experimentally high gradients effects, we chose to work with high \( V_{AC} \) voltages of about 5 V. The other parameters were the same as those used previously in the case of low gradients (\( z = 25 \text{ nm}, R = 50 \text{ nm} \), and \( V_{DC} = 0.5 \text{ V} \)). The experiments were performed with a Bruker (previously Veeco) Enviroscope\textsuperscript{TM} AC phase shifts and amplitude signals of the mechanical phase shift (proportional to the gradient) were recorded at different temperatures from 22 °C to 80 °C. We used a high temperature cantilever holder which incorporates a small heater plate. The primary use of the heater is to eliminate water molecules or volatile components that, evaporating from the hot sample,\(^{16}\) may deposit on the tip and cantilever’s backside. The correspondence between the tip heater voltage and the temperature of the probe was given in the microscope users.

![Figure 1](image-url)
manual, where it is not specified how this calibration was made. We used standard EFM cantilevers (Nanosensors EFM) having a free oscillating frequency (at 22°C) $f_0 \approx 70.04\,\text{kHz}$ and a stiffness (measured by thermal tune) $k_0 \approx 4.1\,\text{N/m}$. The PtIr5 coating is an approximately 25 nm thick double layer of chromium and platinum iridium on both sides of the cantilever.

Typical values of gradient components were calculated to be about $G_0 \approx 5 \times 10^{-2}\,\text{N/m}$, $G_1 \approx 9 \times 10^{-3}\,\text{N/m}$, and $G_2 \approx 2 \times 10^{-2}\,\text{N/m}$, which corresponds to a ratio varying between 5 and 20, according to the gradient component, compared with $V_{AC} = 1\,\text{V}$. Experimental amplitudes of AC gradients are shown in Figure 3. Whatever the component, $\omega_x$ or $2\omega_x$, we observe a quite similar behaviour. At ambient temperature, $Amp_{\omega_x}(f_x)$ (Figure 3(a)) and $Amp_{2\omega_x}(f_x)$ (Figure 3(b)) exhibit a second-order filter behaviour. A large bump appears at low frequency, reaching its maximum at an intermediate frequency and then the amplitude decreases sharply. In the steepest part, the slope is about 40 dB/decade. When the temperature is increased gradually from 22°C to 80°C, the bump disappears progressively both on $\omega_x$ and $2\omega_x$ components. Another effect can be reported at low frequency where the amplitude level increases when the temperature increases. This effect is much more noticeable on the $2\omega_x$ component (Figure 3(b)). Concerning the $\omega_x$ component, the observation of low frequency amplitudes below 40°C was difficult up to 100 Hz because of too low levels; therefore, the curves 22 and 30°C have not been reported in Figure 3(a).

The observations are consistent with the simulations using the general model proposed in Sec. II C 1, whose results are shown in Figure 4. The experimental reduction of the bump with temperature is consistent with a decreasing of the quality factor $Q$ of the cantilever. Such temperature
dependence has been observed on coated cantilevers by Klempner et al.\textsuperscript{12} The decrease of \( Q \) can reach 35\% from 10 to 90 °C. In the simulations, \( Q \) was decreased by 60\% from 330 to 150, and this allowed to explain qualitatively (the order of magnitudes are good) the reduction of the bump on both \( \omega_x \) and \( 2\omega_x \) components, as well as the low frequency effect on amplitude with temperature. We note that the simulation gives higher cut frequencies on the \( \omega_x \) component, as slightly observed experimentally in Figure 3. We also note that the bumps appear more important on simulations than in the experiments. We do not know yet how to explain such a difference. It is quite possible that the model, based on mass-spring approximation, is not complete enough to provide a quantitative approach; nevertheless it is clear that it allows explaining the observed effects.

Experimental phases of AC gradients obtained with same cantilever are shown in Figure 5. They produce a similar effect to the amplitudes, i.e., the presence of a bump at an intermediate frequency, which gradually disappears with increasing temperature. This effect is more pronounced compared to the amplitude. The bump disappears clearly at 60 °C on both \( \omega_x \) (Figure 5(a)) and \( 2\omega_x \) (Figure 5(b)) components. We were initially surprised by such a phase behavior. Indeed, amplitude observations gave the impression that we were in presence of a second order low-pass filter, and as a result, the phase should not present a bump but a transition of \( 180° \) (90° at the cutoff frequency) and becoming less steep as the damping decreases. Therefore, this hypothesis was not conclusive. However, as can be seen in Figure 6, the simulations perfectly describe the observed effect. By reducing the quality factor from 330 to 150, the bump was removed from a threshold value \( Q_{th} = 170 \). We note that this corresponds to a threshold ratio \( Q_{th}/k_0 \approx 42.5 \). Beyond this value, there may be a bump, below should not be any. From an experimental view, we noted a decrease in the quality factor from 253 (at 22 °C) to 185 (at 80 °C), which is very close and consistent with the simulations.

IV. SYNTHESIS ON BUMP APPEARING CONDITIONS: 
\( Q_n/k_n \) DEPENDENCE, CONSEQUENCE ON OSCILLATING MODE, ATMOSPHERE, AND TEMPERATURE EFFECTS

We here propose a generalization of the conditions of appearance of a bump whatever the mode and depending on the intensity of the AC gradient, high or low. We performed simulations based on standard EFM cantilevers that have a fundamental frequency \( f_0 = 70 \) kHz. To take into account the possible dispersion of stiffness values, we considered two cantilevers: A (4 N/m, “stiff case”) and B (2 N/m, “soft case”). Cantilever A is similar to that used in the experiments. Threshold values \( Q_{th} \) and \( Q_{th}/k_n \) for the 3 first modes and at two values of AC voltages (1 V and 5 V) are reported in Table I. They were estimated on the basis of phase simulations. Concerning the fundamental mode, if \( V_{AC} = 1 \) V, for both A and B cases, \( Q_{th} \) is elevated, greater than or equal to 2000. This means that in ambient conditions at room temperature and even by heating the probe, it is impossible to have a bump while under vacuum, typical values of quality factors (\( \sim 30\,000 \)) exceeding threshold values (between 2000 and 3000) a bump may rise. We note that \( Q_{th}/k_0 \) is elevated, and it is nearly constant for two cantilevers around 700. If \( V_{AC} = 5 \) V, thresholds are much lower. This is a possible
TABLE I. Calculated threshold values for bump appearing for two different cantilevers of a same batch, in a scenario of low stiffness (2 N/m) and high stiffness (4 N/m). Our experimental observations would go to the high stiffness case. For a given voltage, $Q_{th}/k_n$ threshold is constant regardless of the vibration mode.

<table>
<thead>
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<th>$V_{AC}$</th>
<th>$n=0$</th>
<th>$n=1$</th>
<th>$n=2$</th>
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<td>$f_n$ (kHz)</td>
<td>$Q_n$</td>
<td>$Q_{th}$</td>
</tr>
<tr>
<td>VAC</td>
<td>$f_0$</td>
<td>22°C</td>
<td>80°C</td>
</tr>
<tr>
<td>1 V</td>
<td>$f_0$</td>
<td>70.3</td>
<td>70.11</td>
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<tr>
<td>5 V</td>
<td>$f_0$</td>
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<td>2000</td>
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</tbody>
</table>

TABLE II. Experimental resonance frequencies and quality factors obtained on first and second vibrating modes at 22°C and 80°C.

<table>
<thead>
<tr>
<th>Air</th>
<th>Vacuum($\approx 1 \times 10^{-4}$ mbar)</th>
<th>22°C</th>
<th>80°C</th>
</tr>
</thead>
<tbody>
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<td>185</td>
</tr>
<tr>
<td>$Q_0$</td>
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<td>3352</td>
<td></td>
</tr>
<tr>
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<td>$f_1$</td>
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<td>442.55</td>
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</tbody>
</table>

To summarize, $Q_{th}/k_n \approx 700$ and 40 for $V_{AC} = 1$ and 5 V, respectively. It is now possible to explain why Portes et al. did not observe any bump, in fact they applied $V_{AC} = 1$ V and their ratio $Q_1/k_1 = 125 < 700$) on the second mode and in vacuum was below the threshold.

In Sec. III, we have shown that by increasing the probe temperature, the bump could disappear, which can be explained experimentally by a decrease of the quality factor with temperature. Table II summarizes experimental data on resonance frequencies and quality factors for $n=0$ and $n=1$ in air and vacuum conditions ($\approx 1 \times 10^{-4}$ mbar) at 22°C and 80°C. It is clear that the resonance frequency decreases with temperature. Such an effect can be interpreted as a decrease of Young’s modulus of the cantilever’s conducting coating layer, as explained in Ref. 14. The temperature coefficient of the Young’s modulus of silicon is $-0.6 \times 10^{-4}$ K$^{-1}$. At the same time, it is more difficult to explain the decrease of the quality factor, observable both in the air and in vacuum and estimated to about 20%–30% whatever the mode. Thermoelastic damping could be responsible for damping effects as mentioned in Ref. 19. In Figure 7, as an example, we show some resonance curves obtained on the second mode, in air and vacuum, at 22 and 80°C. This mode was preferred to the others ($n=0$ and 2) because it showed no asymmetry in the curves. The resonance curves were made comparable by injecting the same energy (same drive amplitude). Under these conditions, we clearly see that the loss of energy is more important at high temperatures.

V. CONCLUSION

In this work we have attempted to interpret the relationship between the magnitude of AC voltage, and a possible bump rising on $\omega_n$ or $2\omega_n$ components of amplitude or phase shifts versus frequency curve, as a function of temperature.

At a constant value of AC voltage, $Q_n/k_n$ ratio appears to be the key element for bump observation and prediction. At low values of AC voltages, because of high $Q_n/k_n$ threshold, ensuring sufficient damping, it is almost impossible to get a bump. At higher AC voltages, the threshold becomes much lower. In such damping conditions, the frequency dependence of force gradients detected in air on the fundamental mode can be strongly affected, as well as in vacuum when using amplitude modulation EFM on the second mode.

FIG. 7. Experimental resonance curves at 22°C and 80°C, in air and in vacuum, using the second mode.
We reported some experimental observations at different temperatures using the fundamental mode in air which were corroborated with simulations of cantilever dynamics in presence of AC gradients. The simulations also allowed predicting bump appearing conditions under vacuum and using higher modes. The threshold ratio $Q_{th}/k_n$ was found to be constant at a given AC voltage. This means that for the same category of cantilevers, given the dispersion of the stiffness, the softer ones will generate bumps at room temperature on the fundamental mode while others will not show such an effect.

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