Temperature dependence of the force sensitivity of silicon cantilevers

U. Gysin,* S. Rast, P. Ruff, and E. Meyer
Klingelbergstrasse 82, CH-4056 Basel, Switzerland

D. W. Lee, P. Vettiger, and C. Gerber
IBM Research Division Zurich, Research Laboratory, CH-8803 Rüschlikon, Switzerland

(Received 2 May 2003; revised manuscript received 2 September 2003; published 8 January 2004)

The resonance frequency ω and internal friction Q^{-1} of the first eigenmode of microfabricated silicon cantilevers are measured in the temperature range of 15–300 K. The analysis shows that variation of Young's modulus is responsible for the temperature dependence of the resonance frequency, whereas the dependence of the geometrical dimensions can be neglected. Accordingly, the data can be fitted by the Wachtman equation, yielding a Debye temperature $\Theta_D = 634$ K. The temperature variation of internal friction Q^{-1} is analyzed in terms of Zener's theory of thermoelastic damping. Due to the temperature dependence of the thermal expansion coefficient α, thermoelastic damping is expected to vanish at 20 K and 125 K. A minimum of internal friction is observed at 20 K, whereas the minimum at 125 K appears to be hidden by other dissipation effects. A maximum of internal friction at 160 K is observed, which is an activation peak due to phonon scattering by atomic-scale defects. The best force sensitivity is achieved at 20 K, where a factor of 10 is gained compared to room temperature.

DOI: 10.1103/PhysRevB.69.045403 PACS number(s): 68.37.Ps, 62.40.+i

I. INTRODUCTION

Ever since force microscopy[1] was invented, cantilevers became important tools to measure small forces of the order of nanonewtons, like those which occur in chemical bonds. Microfabricated silicon cantilevers became the new standard in force microscopy, because probing tips with a small radius of curvature can be integrated on the cantilever in a batch process.[2] Measurements in the dynamic mode, where the cantilever is oscillated at its resonance frequency, showed that the force sensitivity can be further improved into the range of femtonewtons. Additional improvement could be made by cooling the cantilevers to cryogenic temperatures. Stowe *et al.*[3] achieved a force sensitivity of 5.6×10^{-18} N/$\sqrt{\mathrm{Hz}}$ at 4.8 K. Mamin and Rugar[4] could demonstrate subattonewton force sensitivity (0.820×10^{-18} N/$\sqrt{\mathrm{Hz}}$) in the millikelvin regime. This work is motivated by the challenge to measure single spins by magnetic resonance force microscopy[5] (MRFM), with which forces in the attonewton regime will have to be detected.[6] Therefore, there is a need to achieve a fundamental understanding of the mechanisms which determine the force sensitivity of microfabricated cantilevers as a function of temperature.

In this paper, it is shown that the temperature dependence of the minimum detectable force is determined by $\sqrt{T}$, the Young's modulus $E=E(T)$, and the internal friction $Q^{-1}=Q^{-1}(T)$. The temperature dependence of the geometrical dimensions can be neglected. The resonance frequency ω and the internal friction Q^{-1} of the first eigenmode of silicon cantilevers are presented as functions of temperature. The resonance frequency versus temperature data are analyzed with the Wachtman equation, which yields a value of $\Theta_D=634$ K for the Debye temperature in agreement with calorimeter measurements. The internal friction versus temperature data show a rather complex behavior, which is interpreted by Zener's theory of thermoelastic damping. Accordingly, the minimum of internal friction at 20 K is related to the temperature dependence of the thermal expansion coefficient α. A maximum at 160 K is interpreted as an activation peak due to phonon scattering by atomic-scale defects.

The highest force sensitivity is found at $T=20$ K, where a minimum of internal friction is predicted by Zener's theory of thermoelasticity. An increase in sensitivity by a factor of 4 is expected from the explicit $\sqrt{T}$ dependence of the sensitivity when the temperature is reduced from 300 K to 20 K. The measured data provide an increase of sensitivity of a factor of 8 and 11 for two selected examples.

II. THEORY

A. Temperature dependence of minimum detectable forces

The force sensitivity of cantilevers is limited by thermal noise. When operated in the dynamic mode, where the cantilever is oscillated at one of its eigenfrequencies, the high Q factors of crystalline silicon sensors yield optimum performance. The minimum detectable force F_n^{min} of a rectangular cantilever of length L, width w, and thickness t operated at its nth transversal eigenmode is:[7]

$$F_n^{min}=\sqrt{\frac{2k_BTD_n\Delta\omega}{Q_n\pi\omega_n}}, \tag{1}$$

where k_B is the Boltzmann constant and $\Delta\omega$ is the bandwidth of the measurement. In order to achieve the highest possible sensitivity, the cantilever should have a low spring constant D_n, and a high eigenfrequency ω_n, and a high quality factor Q_n and has to be operated at low temperature. Experimentally, it has been shown that operation at higher modes of silicon cantilevers does not improve the force sensitivity.[8] Therefore, we will restrict our discussion to the first eigenmode. The frequency of the first eigenmode of the transversal deflection of a homogeneous beam with uniform cross section $A = wt$ is given by[8,9]

$$\omega_1 = (1.875)^2 \frac{t}{L^2} \sqrt{\frac{E}{12\rho}} \tag{2}$$

and the spring constant is

$$D_1 = \frac{Et^3 w}{4L^3},$$

where ρ is the density of the cantilever.

Thus, both the eigenfrequency ω_1 and the spring constant D_1 depend on the Young's modulus E and the geometrical dimensions of the rectangular cantilever. The temperature dependence of the geometrical dimensions is weak. The difference between 0 K and room temperature is less than 0.5%, which can be neglected in comparison to the temperature dependence of the Young's modulus.

B. Temperature dependence of the Young's modulus

The temperature dependence of the Young's modulus E is due to anharmonic effects of the lattice vibrations. An analytical solution of this challenging problem does not exist. Wachtman *et al.*[10] suggested a semiempirical formula, which is valid for silicon in the high-temperature limit:[11]

$$E(T) = E_0 - BT \exp\left(-\frac{T_0}{T} \right), \tag{3}$$

where E_0 is the Young's modulus at 0 K. The constants $B > 0$ and $T_0 > 0$ are temperature independent. Wachtman *et al.* expected a correlation between T_0 and the Debye temperature Θ_D and between B and the Grueneisen parameter γ. A complete theory for this problem could not be provided at that time. Later, Anderson[12] derived a similar expression [Eq. (3)] for the adiabatic bulk modulus B_S:

$$B_S(T) = B_0 - \frac{\gamma\delta}{V_0} 3RTH\left(\frac{\Theta_D}{T} \right), \tag{4}$$

where

$$H(x) := \frac{3}{x^3} \int_0^x \frac{\xi^3}{e^{\xi} - 1} d\xi,$$

R is the ideal gas constant, δ is the Anderson-Grueneisen parameter, B_0 is the bulk modulus, and V_0 is the volume at 0 K. Note that the product $\gamma\delta$ is assumed to be temperature independent. Equation (4) can be derived from the equation

$$B_S(T) = B_0 - \frac{\gamma\delta}{V_0} \int_0^T C_v dT \tag{5}$$

by the use of the Debye approximation. Equation (5) shows the close relationship between the mechanical property B_S and the specific heat C_V. In this approximation Eq. (5) satisfies Nernst's theorem, where the temperature derivatives of the elastic constants must vanish at 0 K.

The bulk modulus and Young's modulus differ by $E_S / B_S = 3(1 - 2\sigma)$, where σ denotes the Poisson constant. For small and constant $d\sigma/dT \ll 1$ the bulk modulus can be replaced by the Young's modulus $E_S = 3B_S$. Comparison of Eqs. (3) and (4) in the limit of high temperatures results in

$$B = \frac{R\gamma\delta}{V_0} \quad \text{and} \quad T_0 \approx \frac{\Theta_D}{2}. \tag{6}$$

These relations can also be used for the isothermal constants, as they differ from the adiabatic elastic constants by $O(\alpha_V^2)$,[13] where α_V is the volume expansion coefficient.

C. Internal friction

An elastic wave dissipates energy due to intrinsic and extrinsic mechanisms. Some of the extrinsic mechanisms such as air damping can be minimized under ultrahigh-vacuum (UHV) conditions. The intrinsic dissipation mechanisms can be regarded as phonon-phonon interactions. The oscillation of the cantilever corresponds to a time-dependent local stress field. The energy landscape of defects, such as interstitials or vacancies, is changed by this stress field. Instabilities of these defects may occur, where atoms jump from one equilibrium position to another. Part of the strain energy is converted into fast atomic oscillations, which then equilibrate with the phonon bath. This jump from one occupation site to another is related to a characteristic activation energy, which is the energy to overcome from one equilibrium position to the next one. Correspondingly, activation peaks, also called Debye peaks, are observed in damping versus temperature plots. In silicon, previous studies have shown that an activation peak exists around 160 K, where the corresponding activation energy is 0.25 eV. The nature of the defects is not completely clear, but there are indications that it consists of hydrogen interstitials.

Another phonon-phonon interaction is the scattering of acoustic phonons with thermal phonons. Zener[14–16] developed a classical theory for this so-called thermoelastic damping, which is summarized below.

D. Thermoelastic damping

A complex frequency is introduced to describe the dissipation of a classical harmonic oscillator. In close analogy, the energy dissipation of a cantilever can be described by a complex Young's modulus $\tilde{E} = E(1 + iQ^{-1})$.

In the case of an ideal material, stress σ and strain e are related by Hooke's law and are always in phase. No internal friction occurs as long as the Young's modulus is a real number. Zener[14–16] extends Hooke's law with the time derivatives of stress and strain:

$$\sigma + \tau_e \dot{\sigma} = M_R(e + \tau_\sigma \dot{e}),$$

where M_R is the relaxed modulus and τ_e and τ_σ are the relaxation times for the stress and strain.

The extension of Hooke's law leads to inhomogeneities in stress and strain, which can cause temperature gradients in the force sensor. The temperature gradients generate local heat currents which increase the entropy of the cantilever and lead to energy dissipation.

In the following calculation of the energy loss, the x axis is assumed to be perpendicular to the cross section A. Only the stress σ_{xx} due to the vibrations along the cantilever axis is of importance. For small deformations e_{xx}, the increase of the energy per volume is then given by[13]

$$dE = TdS + \sigma_{xx} de_{xx},$$

where $\sigma_{xx} de_{xx}$ is the work performed by the vibrations.

To obtain the internal friction Q^{-1}, Zener[15] calculates the time average of stress σ_{xx}, strain e_{xx} and temperature T. With the temperature distribution given by the law of diffusion and the boundary condition that no heat flow perpendicular to the surfaces of the cantilever, the internal friction is

$$Q^{-1} = \frac{\alpha^2 TE}{C_p} \frac{\omega\tau}{1+(\omega\tau)^2}. \tag{7}$$

The temperature dependence of internal friction is given by an explicit linear dependence and by a implicit dependence of the linear thermal expansion coefficient α, the Young's modulus E, and the specific heat capacity C_p.

The frequency dependence is characterized by the relaxation time $\tau = \sqrt{\tau_\sigma \tau_e}$. For a rectangular cantilever with the thickness t the relaxation time is

$$\tau = \frac{t^2}{\pi^2 D},$$

where D is the thermal diffusion coefficient. For low frequencies $\omega \ll \tau^{-1}$, the vibrations are isothermal and a small amount of energy is dissipated. In the high-frequency range $\omega \gg \tau^{-1}$, the cantilever behaves like an adiabatic system with low-energy dissipation similar to the low-frequency range. At frequencies $\omega \sim \tau^{-1}$ stress and strain are out of phase and a maximum of internal friction occurs.

Another expression for the thermoelastic damping is given by Lifshitz and Roukes:[17]

$$Q^{-1} = \frac{\alpha^2 TE}{C_p} \left(\frac{6}{\xi^2} - \frac{6}{\xi^3} \frac{\sinh \xi + \sin \xi}{\cosh \xi + \cos \xi} \right), \tag{8}$$

where

$$\xi = b \sqrt{\frac{\omega}{2D}}.$$

The temperature dependences of Eqs. (7) and (8) are identical. The frequency dependences in the isothermal range are also identical for both expressions and differ only slightly in the adiabatical range.

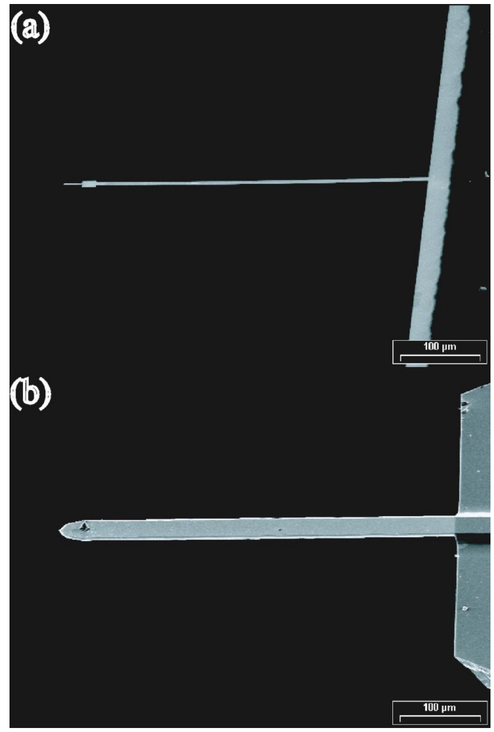

FIG. 1. SEM pictures of the cantilevers. Picture (a) shows the ultrahigh-sensitive cantilever ($L = 454$ μm, $w = 4$ μm, $t = 0.44$ μm, and spring constant 0.1×10^{-3} N/m) made by IBM-Rueschlikon and picture (b) the commercially available NANOSENSORS cantilever (Ref. 18), ($L = 450$ μm, $w = 45$ μm, $t = 2$ μm, and spring constant $D = 0.176$ N/m).

III. RESULTS AND DISCUSSIONS

A. Experimental setup

The microscope is cooled with a flow cryostat (JANIS Research Company ST-400) and can be operated under UHV conditions. The temperature of the cantilever can be varied in the range of $T = 15$–300 K. The deflections of the force sensor are measured with the beam deflection technique.

The resonance frequency $f = \omega/(2\pi)$ of the cantilever is measured with a spectrum analyzer (HP 3589A). The cantilever is excited with a periodic force at the resonance frequency. The amplitude of the cantilever is obtained by filtering the signal with a lock-in amplifier (STANFORD Research Systems SR830). The exponential decay of the signal is used to determine the decay time $\tilde{t}$. The internal friction of the cantilever is then given by $Q^{-1} = 2/(\omega\tilde{t})$.

Figure 1 shows scanning electron microscope (SEM) pictures of two cantilevers which were investigated. Picture (a) shows the cantilever fabricated by IBM-Rueschlikon with a length $L = 454$ μm, a width $w = 4$ μm, a thickness $t = 0.44$ μm, and a spring constant $D = 0.1 \times 10^{-3}$ N/m, and picture (b) shows the commercially available NANOSENSOR cantilever[18] with dimensions $L = 450$ μm, $w = 45$ μm, $t = 2$ μm, and spring constant $D = 0.176$ N/m.

B. Frequency shift

Figure 2 illustrates the temperature dependence of the eigenfrequency f of cantilever (b). The squares are the mea-

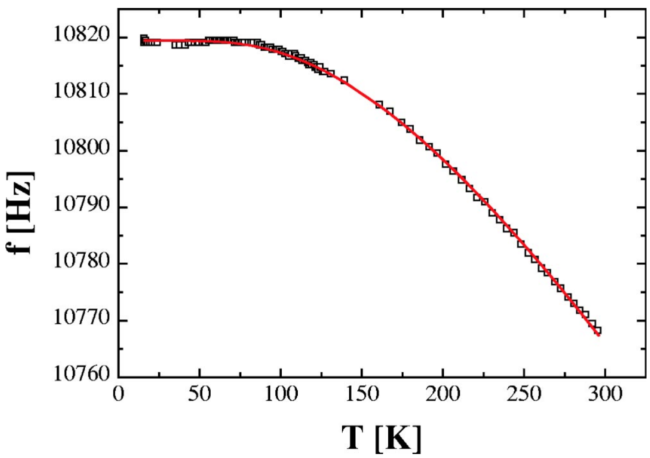

FIG. 2. Measured frequency shift of the cantilever (b) (Ref. 18). The frequency shift is given by the temperature dependence of the Young modulus, as the relative changes of the geometrical dimensions are less the 0.5%. The fitting parameters for the Young modulus give a Debye temperature $\Theta_D = 634$ K.

sured values. The line is the fitting curve for the eigenfrequency using Eq. (2). The Young's modulus in Eq. (2) is derived from expression (3). The fitting parameters $T_0 = 317$ K, $B = 15.8$ MPa/K, and $E_0 = 167.5$ GPa are obtained. The value for the Young's modulus E_0 at 0 K corresponds to the literature value for the [110] direction of silicon.[19] This is the direction parallel to the cantilever's length axis.[9] The measured value for the Debye temperature Θ_D is 634 K. The deviation from the literature value[20] 645 K is less then 2%. The good agreement justifies the approximation $\Theta_D \approx 2T_0$ to be valid in the low-temperature range also. Subsequent experiments with other cantilevers of the type (b) give consistent results with deviations of 10% compared to the above results. Cantilever (a) gives similar values for the elastic modulus, but a reduced value for the Debye temperature of 430 K. Since cantilever (a) is rather thin, the influence of the oxide film is a possible source for these deviations.

A comparison of the specific heat capacity C_v and the negative temperature derivative of the Young's constant $-dE/dT$ is shown in Fig. 3. As predicted by Eq. (5) the graphs should be identical if all simplifying assumptions are legitimate. The good agreement of the curves demonstrates the temperature independence of the product $\gamma\delta$, although the Gruenseisen parameter γ and the Anderson-Grueneisen parameter δ for silicon vary strongly in the low-temperature range.[21,22]

C. Dissipation

Figure 4 shows the measured temperature dependence of the internal friction of cantilever (b).[18] The first eigenmode has a frequency in the isothermal region ($\omega \ll \tau^{-1}$) of thermoelastic damping. Two extrema at 20 K and 160 K are found. The maximum at 160 K is an activation peak and was observed in previous experiments[23,24] for silicon resonators in the kHz and MHz range in the temperature range $T = 120$–200 K. The minimum at 20 K is in agreement with

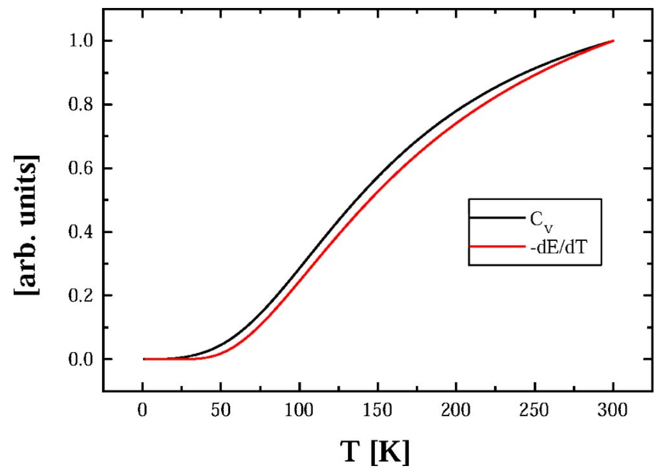

FIG. 3. Comparison of the specific heat capacity C_v and the negative temperature derivative of Young's modulus $-dE/dT$. As required by Nernst's theorem, the temperature deviation of the elastic constants vanishes at 0 K. Both curves are identical if the product of the Grueneisen parameter γ and the Anderson-Grueneisen parameter δ is temperature independent and the volume expansion can be neglected.

Zener's model. The temperature dependence of thermoelastic internal friction in Zener's model is dominated by the thermal expansion coefficient α, as α^2 appears in Eqs. (7) and (8). The value of α for silicon varies by two orders of magnitude from 1 K to 300 K and is even negative between 20 and 125 K. This peculiar behavior of the thermal expansion is experimentally verified[25] and theoretically explained.[22,26] The thermoelastic damping model predicts extrema of internal friction at 20 K and 125 K, which are related to the minima of α^2. Our data (cf. Fig. 4) show the minimum at 20 K. The second minimum at 125 K is not observed, which may be related to additional channels of dissipation. The activation peak at 160 K is relatively strong and broad, which may explain the nonobservation of the minimum at

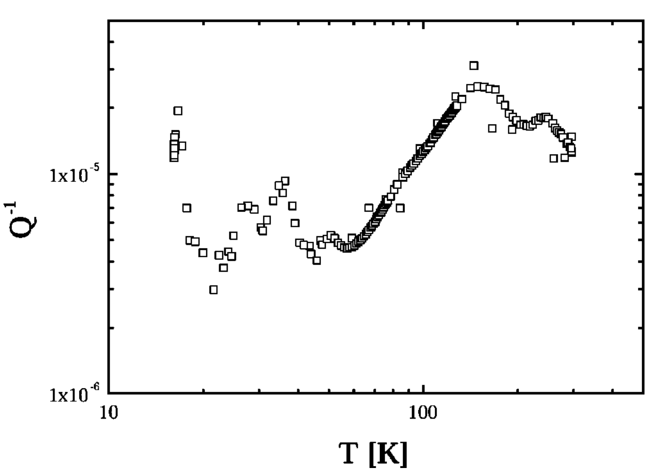

FIG. 4. Measured internal friction of cantilever (b) (Ref. 18). The minimum at 20 K is observed, as predicted by Zener's theory of thermoelastic damping. The peak at $T = 160$ K is an activation peak and has been observed in other experiments (Refs. 23 and 24).

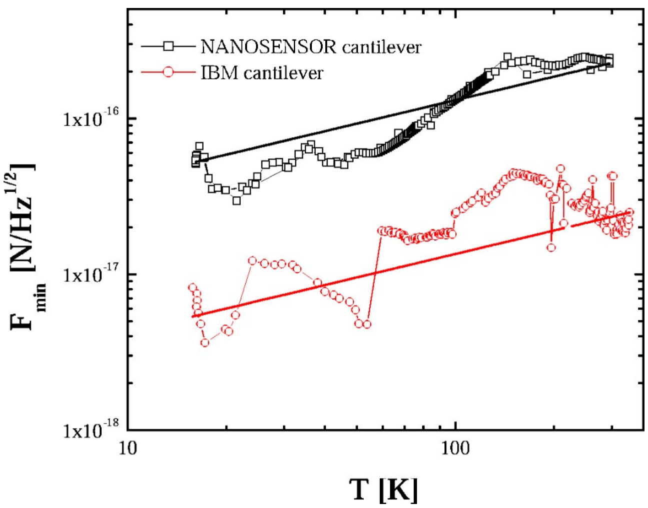

FIG. 5. Minimum detectable force of both cantilevers. The squares and circles are the measured temperature dependences of the sensitivity with variables Q^{-1} and E and the lines are the explicit temperature dependence with constant Q^{-1} and E.

125 K. Other possible explanations may be the dissipation in the thin oxide layer or surface effects.

The resulting minimum detectable force from the measured eigenfrequency f and the internal friction Q^{-1} of both cantilevers is shown in Fig. 5. The squares and circles correspond to the sensitivity with temperature-dependent Young's modulus $E=E(T)$ and internal friction $Q^{-1}=Q^{-1}(T)$, where the lines correspond to the explicit temperature dependence $F^{min}\propto\sqrt{T}$ with constant Young's modulus and internal friction. The discontinuity of the measured sensitivity of the cantilever (a) at $T=60$–70 K is unexplained.

IV. CONCLUSIONS

The temperature dependence of the eigenfrequency of the cantilever is dominated by the variation of the Young's modulus, whereas the temperature dependence of geometrical dimensions due to thermal expansion, especially the thickness, can be neglected. The temperature dependence of the Young's modulus is described by Wachtman's equation (3). A close relationship between Young's modulus and the specific heat is found. The resulting value of the fitting parameters of the Debye temperature is in good agreement with calorimeter measurements.

The understanding of the internal friction properties of silicon is more complex. The observed internal friction is a sum of several damping mechanisms, which are difficult to separate. Generally, internal friction is described as phonon-phonon interaction. Defects in the bulk or on the surface can lead to dissipation. The acoustic wave leads to local changes in the energy landscape of the defects, which may lead to instabilities and energy losses. This part of the internal friction is characterized by activation energy of the defect and leads to activation peaks in the Q^{-1}-vs-T plots. The maximum at 160 K is attributed to such an activation peak, which is in agreement with previous experiments.[23,24] Another phonon-phonon interaction is the thermoelastic relaxation, described by Zener's model. The elastic phonons couple via the linear thermal expansion coefficient α to a bath of thermal phonons. The damping induced by thermoelastic damping of silicon is expected to show two minima at 20 K and 125 K. At these values the linear thermal expansion coefficient α of silicon is zero and no thermoelastic relaxation occurs. The measured data reveal the minimum at 20 K. The second minimum at 125 K appears to be hidden due to other dissipation channels. It is possible that the rather strong and broad activation peak at 160 K dominates, leading to the nonobservation of the 125 K minimum.

Both cantilevers show the highest sensitivity at $T=20$ K, where a minimum of internal friction is predicted by Zener's theory of thermoelasticity. An increase in sensitivity by a factor of 4 is expected from the explicit $\sqrt{T}$ dependence of the sensitivity when the temperature is reduced from 300 K to 20 K. The measured data provide an increase of sensitivity by a factor of 8 for the rectangular cantilever (b) (Ref. 18) and by a factor 11 for the custom paddle cantilever (a). Below 10 K, interesting effects of the temperature dependence of the internal friction are expected, because transport phenomena play an essential role in Zener's theory of thermoelastic damping. For most materials the thermal conductivity vanishes at 0 K and no thermoelastic damping is expected.

ACKNOWLEDGMENTS

This work was supported by the Swiss National Foundation, the "Kommission zur Förderung von Technologie und Innovation," the national program TopNano 21, and the National Center of Competence in Research on Nanoscale Science. We thank J.-P. Ramseyer for the SEM images.

*Electronic address: Urs.Gysin@unibas.ch

[1] G. Binnig, C. Quate, and C. Gerber, Phys. Rev. Lett. **56**, 930 (1986).
[2] T. Albrecht and C. Quate, J. Appl. Phys. **62**, 2599 (1987).
[3] T.D. Stowe, K. Yasumura, T.W. Kenny, D. Botkin, K. Wago, and D. Rugar, Appl. Phys. Lett. **71**, 288 (1997).
[4] H.J. Mamin and D. Rugar, Appl. Phys. Lett. **79**, 3358 (2001).
[5] J.A. Sidles, J.B. Garbini, K.J. Bruland, D. Rugar, O. Zueger, S. Hoen, and C.S. Yannoni, Rev. Mod. Phys. **67**, 249 (1995).
[6] G.P. Berman and V.I. Tsifrinovich, Phys. Rev. B **61**, 3524 (2000).
[7] S. Rast, C. Wattinger, U. Gysin, and E. Meyer, Nanotechnology **11**, 169 (2000).
[8] S. Rast, C. Wattinger, U. Gysin, and E. Meyer, Rev. Sci. Instrum. **71**, 2772 (2000).
[9] U. Rabe, K. Janser, and W. Arnold, Rev. Sci. Instrum. **67**, 3281 (1996).
[10] J.B. Wachtman, W.E. Tefft, D.G. Lam, and C.S. Apstein, Phys. Rev. **122**, 1754 (1961).
[11] M.L. Nandanpawar and S. Rajagopalan, J. Appl. Phys. **49**, 3976 (1978).

[12] O.L. Anderson, Phys. Rev. **144**, 553 (1966).
[13] L. Landau and E. Lifshitz, *Theory of Elasticity* (Pergamon Press, New York, 1986).
[14] C. Zener, Phys. Rev. **52**, 230 (1937).
[15] C. Zener, Phys. Rev. **53**, 90 (1938).
[16] C. Zener, Phys. Rev. **53**, 100 (1938).
[17] R. Lifshitz and M. Roukes, Phys. Rev. B **61**, 5600 (2000).
[18] NANOSENSORS Pointprobes.
[19] L. Boernstein, *Band 17 Halbleiter* (Springer-Verlag, Berlin, 1982).
[20] *Handbook of Chemistry and Physics*, 80th ed. (CRC Press, Boca Raton, 1999–2000).
[21] J. Philip and M.A. Breazeale, J. Appl. Phys. **54**, 752 (1983).
[22] S. Wei, C. Li, and M.Y. Chou, Phys. Rev. B **50**, 14 587 (1994).
[23] S. Evoy, A. Olkhovets, L. Sekaric, J.M. Parpia, H.G. Craighead, and D.W. Carr, Appl. Phys. Lett. **77**, 2397 (2000).
[24] B.S. Berry and W.C. Pritchet, J. Appl. Phys. **67**, 3661 (1990).
[25] K.G. Lyon, G.L. Salinger, and C.A. Swenson, J. Appl. Phys. **48**, 865 (1977).
[26] S. Biernacki and M. Scheffler, Phys. Rev. Lett. **63**, 290 (1989).