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Sensitivity optimization of the scanning microdeformation microscope and application to mechanical characterization of soft materials

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In this article we present the study of the sensitivity optimization of our system of micromechanical characterization called the scanning microdeformation microscope. The flexural contact modes of vibration of the cantilever have been modeled. We discuss the matching between the cantilever stiffness and the contact stiffness which depends on the sample material. In order to obtain the best sensitivity, the stiffnesses must be the closest one to each other. Because the length of the cantilever directly affects its stiffness, the cantilever geometry can be optimized for different materials. We have validated this study with measurements on a soft material the polydimethylsiloxane with a cantilever optimized for materials of Young's moduli of some megapascals. Experimental results obtained with two different samples have shown the high sensitivity of the method for the measurement of low Young's moduli and have been compared with nanoindentation and dynamic mechanical analysis results. © 2008 American Institute of Physics. [DOI: 10.1063/1.2894208]

I. INTRODUCTION

Over the past few years, the atomic force microscope (AFM) has been used not only for imaging topography but also to probe mechanical properties at nanometre scale. Presently, by using force modulation mode measurements, elastic properties of many materials can be estimated.¹⁻⁷ Our technique called scanning microdeformation microscope⁸ (SMM) works on higher scale (mesoscopic scale) and gives local mechanical spectroscopy for mechanical characterization. We present in this article the use of the SMM to characterize a soft material—polydimethylsiloxane (PDMS)—which is a viscoelastic polymer even used for microfluidic applications. We made a detailed study of the sensitivity of the system in order to optimize the cantilever sizes related to the stiffness of the measured sample. And we validated it by characterizing two different PDMS samples.

II. THE SCANNING MICRODEFORMATION MICROSCOPE

The SMM is a kind of ac-force contact microscope. The sensor is a micromechanical resonator (Fig. 1) composed of a silicon cantilever with a small sharp sapphire tip at the end. The cantilever is glued onto a piezoelectric bimorph transducer at the other end. The transducer excites the vibration of the tip-sample system. The tip remains in contact with the sample and vibrates at some kilohertz with an amplitude of some nanometers. Amplitude and phase of the vibrating cantilever are measured with a high sensitivity heterodyne interferometer.^{9,10} The operation of the SMM is described in Fig. 2.

The signal at the probe output is averaged with the double-phase lock-in amplifier. Amplitude and phase of the cantilever displacement are recorded by the computer. More-

over, a modulated laser diode is used in a deflectometer to control the static force applied on the sample thanks to the second lock-in (and the third one can be used for a transmission mode operation). This microscope is an effective tool to image surfaces and subsurfaces with heterogeneous local elasticity or to characterize elastic properties of a material. In the framework of this study, we use it to measure Young's moduli. So we put the tip in contact with the sample and we apply an additional static force by vertically displacing the clamped end of the cantilever. Then we scan the excitation frequency. The resonant frequency depends on the static force applied via the contact stiffness. Actually, by measuring this resonant frequency, we can estimate local contact stiffness and then, with a well-suited model, the local Young's modulus.

Other ultrasonic noninvasive methods such as atomic force acoustic microscope, ultrasonic force microscopy, or AFM spectroscopy with heterodyne interferometer make such a characterization on the nanometer scale but with less accuracy because the contact model must take into account additional forces on this scale.^{1-3,7} We can also notice the nanoindentation and particularly continuous stiffness measurement technique which is a destructive method which enables local elasticity measurements.¹¹

III. MODEL

We have used a continuous model^{6,12} (Fig. 3) to obtain Young's moduli values of tested samples from the measured contact resonant frequencies. The cantilever is represented as a beam interacting with the sample through two springs k_N and k_T . The piezoelectric bimorph transducer action on the cantilever has been modeled as simple mass m_p and spring k_p .

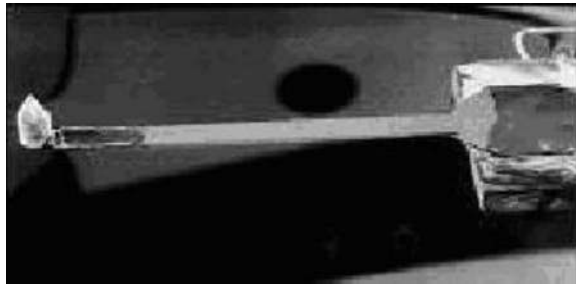


FIG. 1. Vibrating sensor of the SMM.

We need to precisely know contact conditions to relate model springs to local elasticity of the sample. Hertz theory of static contact provides the relation between the static force applied and the contact effective Young's modulus E^* ,¹³

$$F = \frac{4}{3}E^*R^{1/2}\delta^{3/2}, \quad (1)$$

with

$$\frac{1}{E^*} = \frac{1 - \nu_t^2}{E_t} + \frac{1 - \nu_s^2}{E_s} \approx \frac{1 - \nu_s^2}{E_s}, \quad (2)$$

$E_t \gg E_s$ (for us $E_{\text{sapph.}} \gg E_{\text{PDMS}}$) where δ is the elastic indentation in the sample and R the radius of the tip. E_t , ν_t and E_s , ν_s are, respectively, Young's modulus and Poisson ratio of the tip and the sample. Thus the static force applied on the sample is related to the static vertical displacement of the cantilever and to the longitudinal stiffness by the following expression:

$$F = \frac{k_c k_N}{k_c + k_N} \Delta z. \quad (3)$$

Mindlin theory on the contact between a sphere and a plane¹³ makes possible to take into account the lateral stiffness and gives the relation between the longitudinal and lateral stiffness,

$$k_T = 4k_N \frac{G^*}{E^*}, \quad (4)$$

with

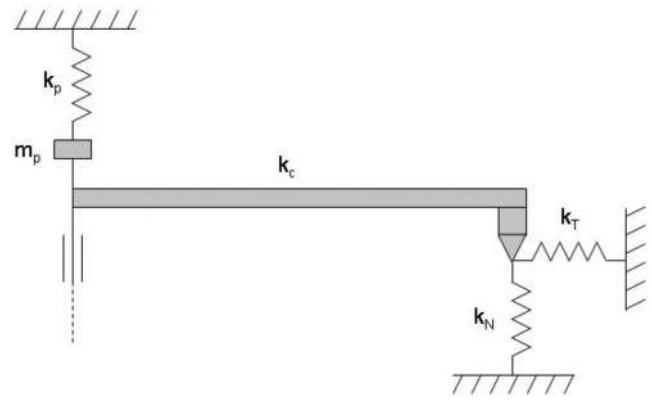


FIG. 3. Model used to describe the behavior of the SMM.

$$G^* = \left[\frac{2(1 + \nu_t)(2 - \nu_t)}{E_t} + \frac{2(1 + \nu_s)(2 - \nu_s)}{E_s} \right]^{-1}, \quad (5)$$

$$k_T \approx \frac{2(1 - \nu_s)}{(2 - \nu_s)} k_N. \quad (6)$$

Let us now consider a small variation of the elastic indentation induced by the harmonic force like Mahaffy *et al.* did,¹⁴

$$\delta = \delta_0 + d\delta, \quad (7)$$

$$F = F_0 + dF. \quad (8)$$

Equation (1) becomes with a first order approximation,

$$F = \underbrace{\frac{4}{3}R^{1/2}E_0^* \delta_0^{3/2}}_{F_0} + \underbrace{2R^{1/2}E_1^* \delta_0^{1/2} d\delta}_{dF}, \quad (9)$$

with E_0^* the static reduced Young modulus of the sample and E_1^* the dynamic reduced Young modulus at the working frequency (which are different for polymers).

The dynamic contact stiffness equals, by definition,

$$k_N = \frac{dF}{d\delta} = 2R^{1/2}E_1^* \delta_0^{1/2}. \quad (10)$$

And finally, using Eqs. (9) and (10),

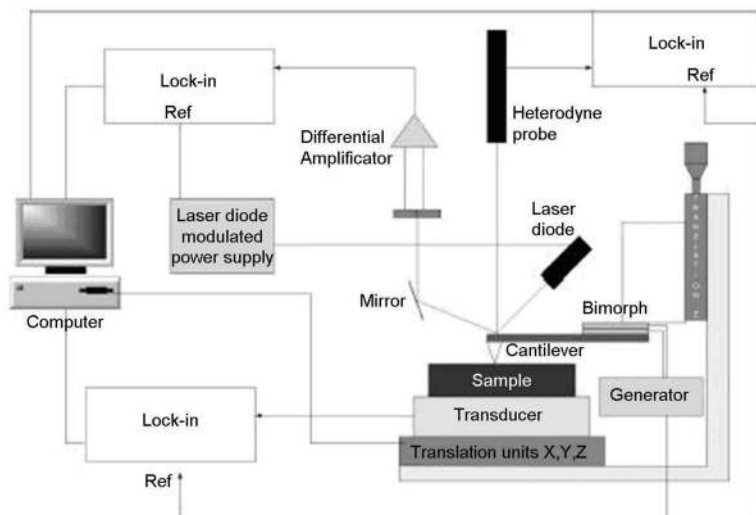


FIG. 2. Principle of the SMM.

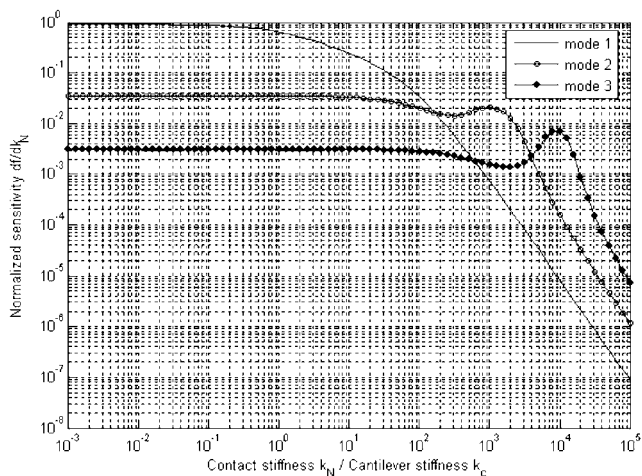


FIG. 4. Normalized flexural sensitivity df/dk_N as a function of contact stiffness k_N (normalized by the cantilever stiffness k_c), with $k_T=0.68 k_N$ for a cantilever with a length of 4 mm, a width of 400 μm , and a thickness of 150 μm for the first three modes.

$$k_N = E_1 \left(\frac{6RF_0}{E_0^*} \right)^{1/3}. \quad (11)$$

To extract Young's modulus from the contact resonant frequency, we solved the linear differential equation for the vibration of the beam with boundary conditions,¹²

$$E_c I \frac{\partial^4 y}{\partial x^4} + \rho S \frac{\partial^2 y}{\partial t^2} = 0, \quad (12)$$

where E_c is Young's modulus, I the area moment of inertia, ρ the volume density, and S the cross section of the cantilever.

IV. SENSITIVITY STUDY

The sensitivity of our measurement system can be defined as $\partial f / \partial k_N$ or $\partial f / \partial E$ which represents the variation of resonant frequency for a variation of contact interaction or local elasticity. Actually, we need to obtain the greatest shift frequency for two materials of different Young's moduli. Such considerations have already been treated for AFM in force modulation by Chang,¹⁵ Wu *et al.*,¹⁶ Turner and Wiehn.^{15,17} For all the sensitivity study we considered that the beam is clamped because the spring k_p modeling the bimorph interaction depends on the cantilever and cannot be applied here. We plotted the normalized sensitivity of the first three flexural modes versus contact stiffness for a beam with a length of 4 mm and with $k_T=0.68k_N$ (Fig. 4). We can see that for soft materials, the first mode is the most sensitive. But when contact stiffness increases and reaches nearly a hundred times the cantilever stiffness, the second mode becomes the most sensitive. And for larger values of contact stiffness the third mode becomes the most sensitive too. We can also notice that the first mode becomes always less sensitive when the contact stiffness is greater, whereas for the other modes the sensitivity first decreases and increases again to reach a local maximum before decreasing with the contact stiffness.

We can also plot the following expression: $S_N = (\partial f / \partial k_N)(k_N/f)$ which represents better the ability to distin-

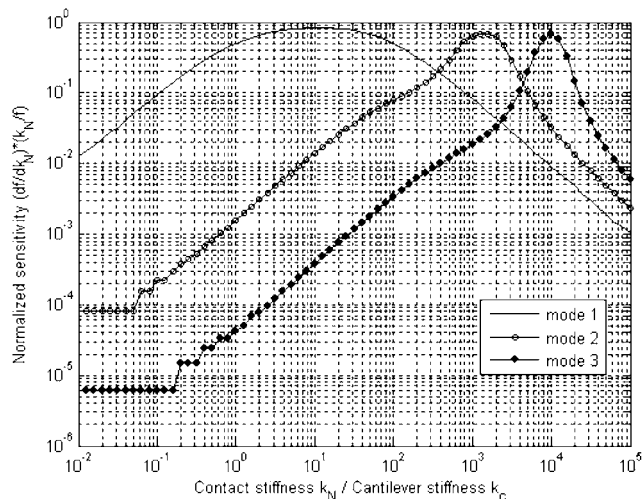


FIG. 5. Normalized flexural sensitivity $(df/dk_N) \times k_N/f$ as a function of contact stiffness k_N (normalized by the cantilever stiffness k_c), with $k_T=0.68 k_N$ for a cantilever with a length of 4 mm, a width of 400 μm , and a thickness of 150 μm for the first three modes.

guish two different materials with Young's moduli close to each other than sensitivity does. Actually S_N is well appropriate because it takes into account the working frequency and contact stiffness. S_N has been plotted for a cantilever with a length of 4 mm versus contact stiffness (Fig. 5). We can see that the curves are different from those of the sensitivity. S_N has a global maximum, whereas precedent sensitivity always decreases with contact stiffness for the first mode. Besides curves appear quite symmetrical on each side from this maximum. By means of this parameter, we highlight precisely the contact stiffness which maximizes the ability to measure elastically close materials. For the first mode S_N reaches a maximum for a contact stiffness of nearly ten times the cantilever's one, 1000 times for the second mode, and 10 000 times for the third mode. We can also notice that the range of high value of S_N is large for the first mode but is reduced for the second mode and even more for the third one.

In order to have the best sensitivity, the cantilever stiffness k_c and the contact stiffness k_N must be close. In fact, if k_N is far bigger than k_c , the cantilever will totally bend. Whereas if k_c is far bigger than k_N , the tip will indent the sample. The cantilever stiffness k_c equals $3E_c I/L^3$, E_c being Young's modulus of the cantilever material, and I the area moment of inertia ($I=bh^3/12$ for a rectangular section beam, b being the width of the beam, and h the thickness). Obviously, the parameters which most affect the stiffness are the length and the thickness of the beam because they are cubed in the expression of k_c . Theoretically, the effect of other parameters such as b , R , or the tip length are negligible for this application, but no generalization is allowed. So we have only focused our study on the length of the cantilever (it is easier and faster to fabricate on the same wafer beams of different lengths than different thicknesses by clean room techniques). We made the sensitivity study for a static force of 0.5 mN. Normalized first flexural mode sensitivity $S_N=(\partial f / \partial E)(E/f)$ is plotted for beam lengths from

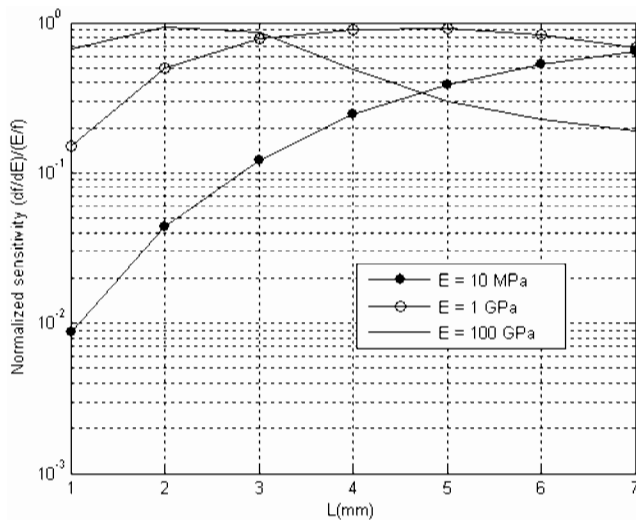


FIG. 6. Normalized flexural sensitivity $(df/dE) \times E/f$ for a cantilever with a thickness of $150 \mu\text{m}$, width of $400 \mu\text{m}$ for the first contact mode (with a static force of 0.5 mN) as function of the length of the cantilever for different Young's moduli of the sample.

1 to 7 mm and materials of Young's moduli of 10 MPa, 1 GPa, and 100 GPa (Fig. 6). Thickness is assumed to be $150 \mu\text{m}$ and width of $400 \mu\text{m}$.

We can notice that, depending on Young's modulus, sensitivity is increasing or decreasing with the length of the cantilever. Actually for a hundred-gigapascal Young's modulus material, the best sensitivity is obtained with a length of 2 mm, whereas for a ten-megapascal Young's modulus one, it is with the length of 7 mm. So the cantilever with a length of 7 mm is optimized to characterize very soft materials. In fact with this cantilever, contact stiffness with Young's moduli of some tens of gigapascals, such as silicon or silica, k_N ($\approx 150\,000 \text{ N/m}$) is nearly 1000 times greater than k_c ($\approx 150 \text{ N/m}$).

So S_N is a very useful parameter to compare the efficiency of our measurement system for different materials.

V. EXPERIMENTAL RESULTS

We tried to validate this precedent study by characterizing a very soft material by the cantilever with a length of 7 mm. A sapphire tip with a length of 0.7 mm and a radius of curvature of $45 \mu\text{m}$ was used. We chose PDMS. PDMS is a silicon-based viscoelastic polymer. Mechanical properties of this material vary with preparation conditions. Actually Young's moduli values can fluctuate in the range of 100 kPa to some megapascals depending on this preparation.¹⁸

We used two different PDMS samples with thicknesses of some millimeters prepared in different conditions and different aging times. To characterize PDMS we put the spot of the laser at the end of the cantilever because it is where the amplitude of vibration of the first contact mode is the greatest (whereas for harder materials the maximum is on the middle of the beam). The model agrees with these observations (see Fig. 7). We can also notice that for hard materials the bimorph interaction spring k_p has a real influence on the modulus computed and has to be fitted with a known sample, whereas with PDMS the value of k_p doesn't hardly change

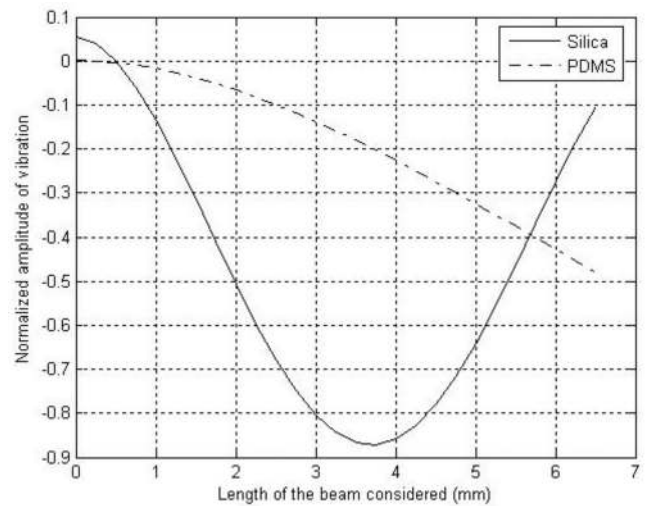


FIG. 7. Theoretical deformation shapes of the first flexural mode in contact with SiO_2 and PDMS.

the result. Figure 8 shows resonances on the first sample of PDMS for different static loads. We can observe the shift frequency and the amplitude losses versus the force because of damping, whereas with an elastic material such as silicon we observed that amplitude increases with the force.

To estimate Young's modulus of the sample we realized 15 successive measures in the same conditions. Static force applied was $150 \mu\text{N}$ because this load provides the best sensitivity (the best slope of frequency versus force). A new contact was obtained for each measurement and we recorded the magnitude spectrum. The dispersion of amplitude is nearly 0.75 \AA and 80 Hz in frequency. So we obtain a mean value for the frequency close to 4.18 kHz. And thanks to the model by taking 0.48 for ν , we computed Young's modulus of 3.4 MPa. ($\pm 0.3 \text{ MPa}$ by considering sensitivity and frequency dispersion). We took 1.7 MPa for the static Young modulus (dynamic mechanical measurement value).

We did the same for our second PDMS sample, and we finally measure a mean resonant frequency of 4.53 kHz and

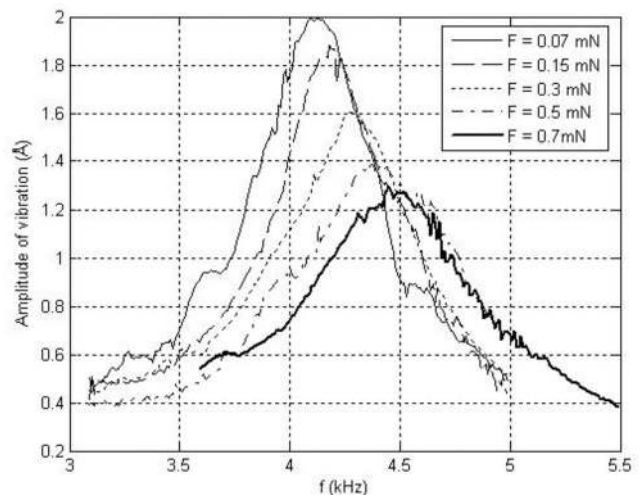


FIG. 8. Experimental spectra of amplitude of vibration (first mode) as a function of frequency in contact with the first PDMS sample for a driving voltage of the bimorph of 0.5 V and for different static forces.

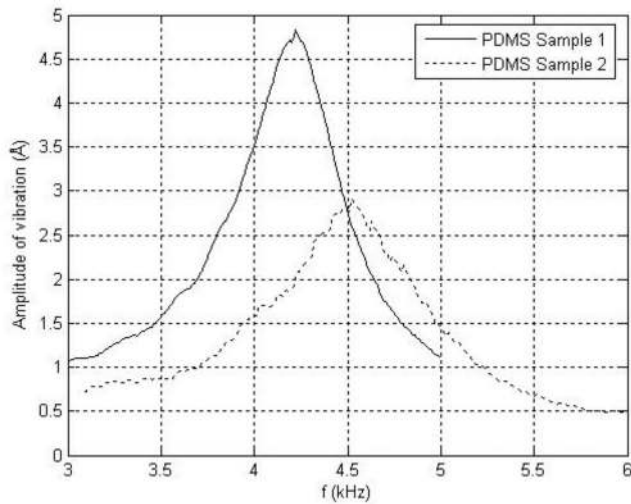


FIG. 9. Experimental spectra of amplitude of vibration (first mode) as a function of frequency in contact with the two different PDMS samples for a static force of $150\mu\text{N}$ and for a driving voltage of the bimorph of 1 V.

also for Young's modulus a value of 5.5 MPa (± 0.3 MPa). We took 2.8 MPa for the static Young modulus (dynamic mechanical measurement value). The SMM has already been tested on standard hard materials such as silicon and silica^{6,12,19} and led to a precision of nearly 5% with the model we are using. We are able to characterize two very soft samples with Young's modulus difference of some megapascals. The shift frequency difference between the two materials is 350 Hz (see Fig. 9). For example, the shift frequency difference with the same cantilever between silica (72 GPa) and silicon (100) (130 GPa) is nearly 1 kHz (see Table I). Experimentally the sensitivity has increased by a factor of 10 000. S_N also has increased by a factor of 3.

We compared our measures with other ones made by two different techniques—nanoindentation (static and continuous stiffness method) and dynamic mechanical analysis—(DMA)—on the same samples for a wide range of frequencies (Fig. 10). DMA measures were conducted on a BOSE electroforce 3200 machine. Sample useful length was 33 mm, for a section of $13.5 \times 3.9 \text{ mm}^2$. A feedback on the position with a peak to peak amplitude of 0.5 mm (corresponding to a deformation of $\pm 7.6 \times 10^{-3}$) for a preload deformation of 7.6×10^{-3} was realized. The sample also was always in traction even at the down point of the cycles. Temperature was nearly 22°C . We can observe that Young's modulus increases with the working frequency which is typical of the evolution of viscoelastic materials. We also notice that SMM measures are a possible continuity at higher frequencies of DMA and nanoindentation curves. Actually the

TABLE I. Frequency shifts and sensitivities for stiff and flexible materials with a cantilever with a length of 7 mm.

| Materials | SiO ₂ /Si | Different PDMSs |
|--------------------------|----------------------|----------------------|
| Δ shift frequency | 1 kHz | 350 Hz |
| Sensitivity | 0.015 Hz/MPa | 167 Hz/MPa |
| S_N | 47×10^{-3} | 136×10^{-3} |

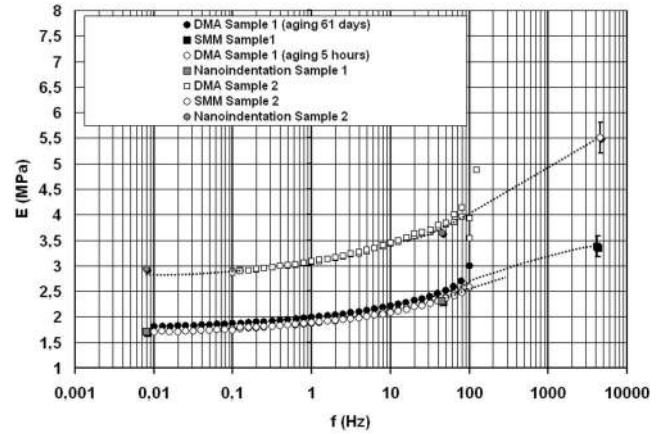


FIG. 10. Young's modulus measured by nanoindentation, DMA, and SMM techniques as a function of the working frequency.

two moduli measured, thanks to the SMM, seem to prolong correctly the curves, but it is difficult to be more affirmative because of the two decades between the measures.

VI. CONCLUSION

In this paper we discussed the optimization of our SMM by taking into account the sample material considered and the stiffness of the cantilever. We can summarize our conclusions as follows.

- (1) The sensitivity of the three first contact modes of the SMM has been studied. Sensitivity decreases with the contact stiffness. The first mode is the most sensitive but when contact stiffness increases higher modes become the most sensitive.
- (2) The parameter S_N (sensitivity reduced to working contact stiffness and frequency) shows for each mode a maximum corresponding to a contact stiffness. Actually we saw that S_N is maximum when the contact stiffness and the cantilever stiffness are of the same order of magnitude for the first mode.
- (3) The sensitivity is also depending on the stiffness of the cantilever. So the length of the cantilever which directly affects its stiffness is a mean to optimize the cantilever with the considered material. We chose lengths of 7 mm because they are optimized for soft materials (increasing sensitivity by a factor of 10 000 and factor of 3 for S_N , compared with hard materials).
- (4) Measures have been made to validate our study. We characterized two different PDMS samples by measuring a shift frequency which enables us to compute their Young's moduli (3.4 and 5.5 MPa) and we obtained a great sensitivity. Comparisons with DMA and nanoindentation measures show a possible continuity between the three techniques.

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