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Generating and measuring the anisotropic elastic behaviour of Co thin films with oriented surface nano-strings on micro-cantilevers

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Abstract

In this research, the elastic behaviour of two Co thin films simultaneously deposited in an off-normal angle method was studied. Towards this end, two Si micro-cantilevers were simultaneously coated using pulsed laser deposition at an oblique angle, creating a Co nano-string surface morphology with a predetermined orientation. The selected position of each micro-cantilever during the coating process created longitudinal or transverse nano-strings. The anisotropic elastic behaviour of these Co films was determined by measuring the changes that took place in the resonant frequency of each micro-cantilever after this process of creating differently oriented plasma coatings had been completed. This differential procedure allowed us to determine the difference between the Young's modulus of the different films based on the different direction of the nano-strings. This difference was determined to be, at least, the 20% of the Young's modulus of the bulk Co.

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Introduction

The study of the elastic and mechanical properties of thin films is of interest in basic and applied research because thin films are used extensively in micro-electronic and micro-electromechanical systems. Because the elastic constants of thin films are different from those of bulk material of the same composition, the elastic constants of the bulk material cannot be used to design thin film devices. Consequently, it is very important to accurately determine the elastic constants of thin films. These properties can be studied using a wide variety of techniques, including the analysis of the substrate curvature [1], micro-beam testing [2], micro-tensile testing [3], cantilever-bending resonance [4], nano-indentation [5], Rayleigh-wave velocity measurements [6] and Brillouin scattering [7]. Among others, Young's modulus is an important parameter for thin-film technological applications.

Micro-cantilevers (MCLs) are mechanical devices with attractive applications; for instance, they are widely used as high-sensitivity sensors in different physical, chemical and biological technologies [8,9]. Another use of MCLs

is in the study of the mechanical properties of thin films [10]. This type of analysis is possible because of the relation between the resonant frequency of MCLs and Young's modulus. If a MCL is coated with a thin film, a change results in the resonant frequency. By measuring this change, one can compute the Young's modulus of the thin film deposited on the MCL.

We conducted a study that demonstrated that the off-normal pulsed laser deposition (PLD) technique allows the simultaneous growth and sculpting of soft magnetic nano-strings with an orientation that is perpendicular to the incidence plane of the plasma and a medium width that can be selected between 8 and 30 nm by selecting an off-normal angle and the appropriate deposition time [11]. Uniaxial in-plane magnetic anisotropy was then generated in the films that would have a value between 10^3 and 10^4 J/m³, depending on the deposition parameters [11]. In addition to magnetic anisotropy, these nano-scale patterned Co films also presented controlled electrical, optical [12] and mechanical anisotropies [13]. In an extension of the study, MCLs were coated with these magnetic nano-strings so that their magneto-mechanical properties were analysed [14].

In this study, we produced Co nano-strings over Si MCLs, validating a differential method of studying the

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elastic anisotropy of these Co thin films in connection with their nano-string morphology. This technique allowed us to determine the difference between the Young's modulus of the films depending on their nano-string direction.

Experimental procedures

Si MCLs, $450 \times 50 \times \approx 2 \mu\text{m}^3$ were coated with Co using PLD via an off-normal-incidence plasma procedure. A Nd:YAG laser beam ($\lambda = 1054 \text{ nm}$, 20-Hz repetition rate, 240 mJ per 4.5-ns pulse, $\approx 12 \text{ GW}$, target spot area $\approx 12 \text{ mm}^2$) was driven onto a pure, polished Co target located inside a chamber with a base pressure of 10^{-6} mbar . The target rotated at 32 rpm and angle of the laser beam from normal to the target was 45° . The MCLs were positioned at a distance of 73 mm from the target and were placed on the lateral surface of a cone with an angle of $\pi - 2\theta$; the axis of the cone was parallel to the direction of the plasma to allow deposition at an off-normal angle, θ , as shown in Figure 1. In this study, the plasma generated reached two MCLs at an off-normal angle of $\theta = 55^\circ$. The cone rotated around its axis at 73 rpm. MCL holders were designed to allow the simultaneous off-normal coating of two MCLs, one parallel (PA-MCL) and one perpendicular (PE-MCL) to the generatrix of the cone, as shown in Figure 1b. Each MCL was located at each end of the diameter of a circle, a circular section perpendicular to the cone axis. Due to the cone rotation and the position of the two MCLs, the MCLs travelled through the plasma in exactly the same circumference, which ensured that each was coated with the same amount of material.

This designed, homemade device allowed the incidence plane of the plasma to be parallel or perpendicular to the longitudinal direction of each MCL. Therefore, the nano-strings generated in the off-normal deposited film were perpendicular (transverse) or parallel (longitudinal) to the longitudinal direction of each MCL, as shown in the right part of Figure 1b. In addition, two glass circles that were 7 mm in diameter were situated on the cone's lateral surface in the same circumference of the two MCLs. This made it possible to perform magnetic measurements.

The two MCLs were selected after the resonant frequency of each, ν_0 , had been determined. The two MCLs were similar because of their equal dimensions and because we did not allow differences between the frequencies of the two selected MCLs higher than 20 Hz in $\approx 10000 \text{ Hz}$. The two MCLs were simultaneously coated with Co in consecutive processes, either with the same coating time or with different coating times, whereas the rest of the parameters remained unchanged.

The same device was used to coat two MCLs with Au under the same conditions, which ensured that our

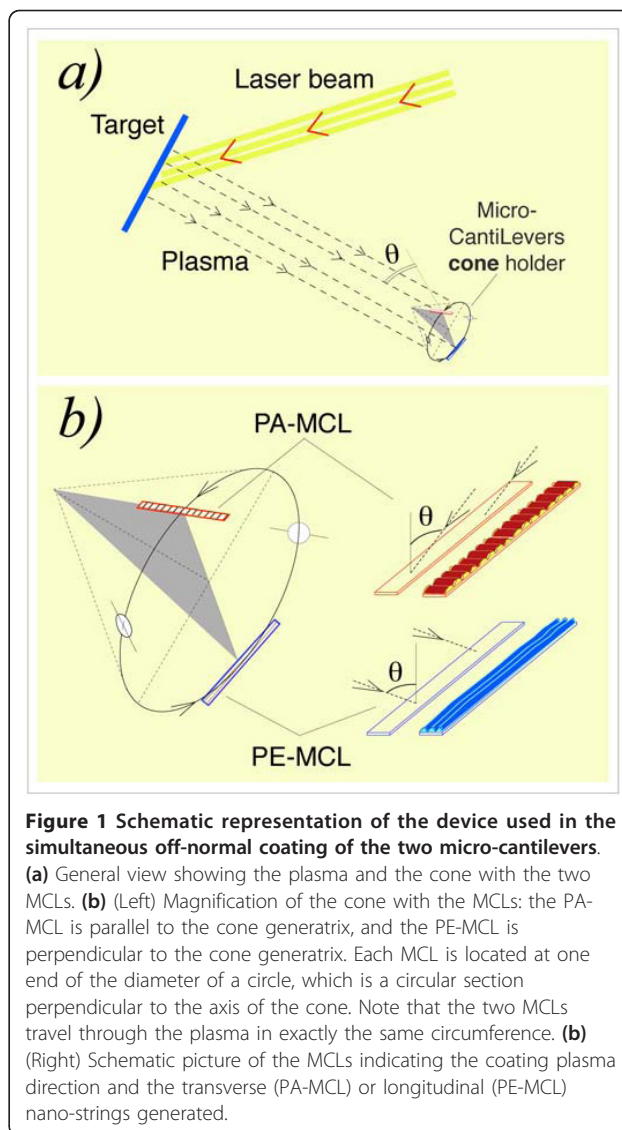


Figure 1 Schematic representation of the device used in the simultaneous off-normal coating of the two micro-cantilevers. (a) General view showing the plasma and the cone with the two MCLs. (b) (Left) Magnification of the cone with the MCLs: the PA-MCL is parallel to the cone generatrix, and the PE-MCL is perpendicular to the cone generatrix. Each MCL is located at one end of the diameter of a circle, which is a circular section perpendicular to the axis of the cone. Note that the two MCLs travel through the plasma in exactly the same circumference. (b) (Right) Schematic picture of the MCLs indicating the coating plasma direction and the transverse (PA-MCL) or longitudinal (PE-MCL) nano-strings generated.

device coated the two MCLs with the same amount of material.

The mechanical resonant frequency of the MCLs, ν_0 prior to coating and $\nu_{(C-MCL)}$ after coating, was determined through location as the working MCL in the head of an atomic force microscope (AFM) [15]. The system performed a driving frequency scan for mechanical oscillation of the MCL, measuring the amplitude and the phase of the MCL's deflection. In this way, the MCL's resonant frequency, ν , was determined. The accuracy of the ν measurements was $\pm 1/10000$.

Scanning tunnelling microscopy (STM) was performed to image the surface morphology of the coated glass circles and also the coated MCLs.

The magnetic hysteresis loops of the coated glass circles were determined using a vibrating sample magnetometer [14]. The value of the measured magnetic

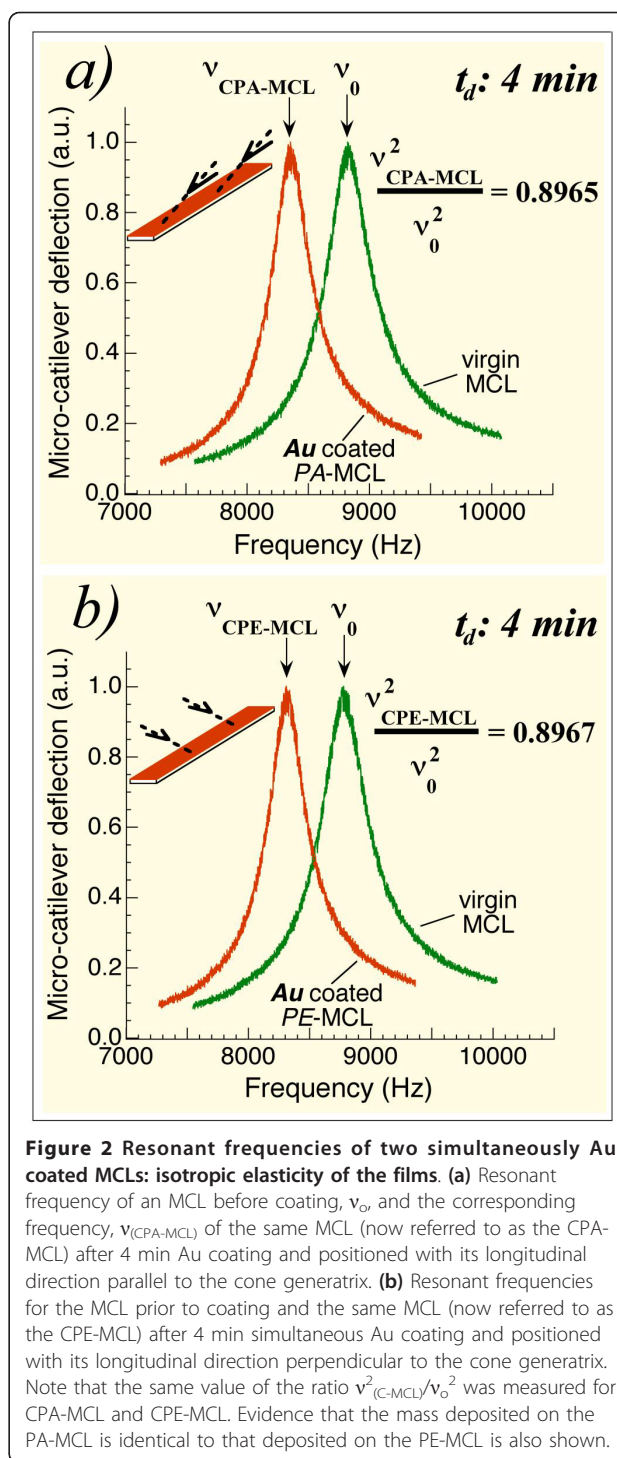
moment of each film was used to deduce its thickness. A deposition rate of ≈ 1.02 nm/min was used in this study. The different films had thicknesses between 0.25 and 28 nm.

Results and discussion

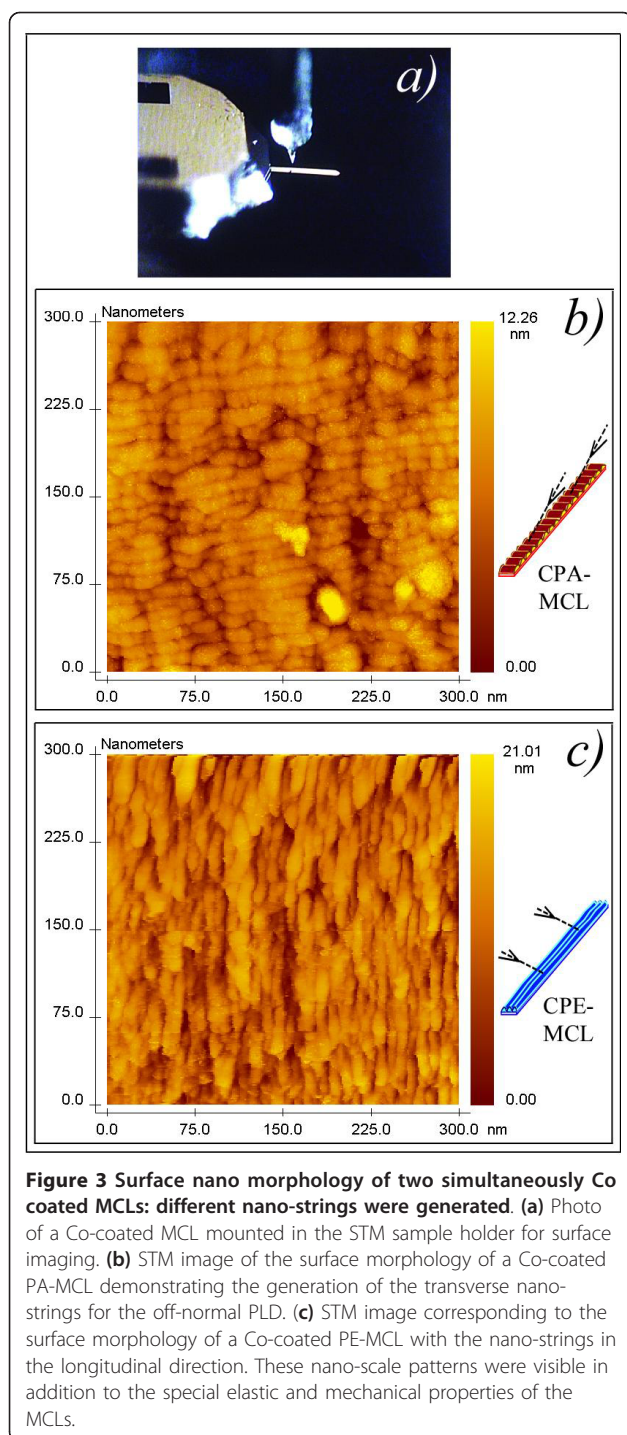
Our previous studies of the surface morphology and physical properties of off-normal PLD Au thin films showed that no nano-strings, no electrical anisotropy and no optical anisotropy were generated in these samples. These results were different from those of off-normal PLD Co. Figure 2 shows the results for the two MCLs simultaneously coated with Au using deposition time $t_d = 4$ min. The resonant frequencies of the MCLs before they were coated with Au, ν_0 , and afterwards, $\nu_{(C-MCL)}$, are indicated in this figure. The resonant frequency of a MCL before coating satisfies the expression $\nu_0^2 \sim k_0/m_0$ with k_0 the spring constant of the MCL and m_0 its mass. For the coated MCL, the C-MCL, the ratio $\nu^2_{(C-MCL)}/\nu_0^2 = (k_{(C-MCL)}/m_{(C-MCL)})/(k_0/m_0)$ will vary when k or m changes: an increase in mass will decrease this ratio, and an increase in the spring constant will increase this ratio. For the PA-MCL, Figure 2a shows the difference between its resonant frequency, ν_0 , and its frequency after coating with its longitudinal direction parallel to the cone generatrix, frequency $\nu_{(CPA-MCL)}$. It is apparent that resonant frequency changes after coating, and the value of $\nu^2_{(CPA-MCL)}/\nu_0^2$ is 0.8965. Figure 2b shows the corresponding results for the PE-MCL positioned with its longitudinal direction perpendicular to the cone generatrix. The corresponding frequency ratio is $\nu^2_{(CPE-MCL)}/\nu_0^2 = 0.8967$. The measurements indicate that this ratio is equal for the two simultaneously Au-coated MCLs; the same shift in resonant frequency was detected. These first results suggest that no mechanical anisotropy was induced in the Au off-normal coated MCLs. Also, important evidence emerged indicating that the mass deposited on the PA-MCL was identical to that deposited on the PE-MCL. This last fact confirms that our system allows differential studies for both MCLs.

The results for the off-normal Co-coated MCLs are different to those for the Au-coated MCLs. Figure 3b shows the surface morphology of a Co-coated PA-MCL, demonstrating the generation of the transverse nano-strings. Figure 3c shows the surface morphology of a Co-coated PE-MCL with longitudinal nano-strings. The average width of the nano-strings was 12 nm. This nano-scale patterned was correlated with the elastic and mechanical properties of the MCLs, as shown in the next results.

The top of Figure 4a shows the resonant frequencies of the PA-MCL: ν_0 , before the coating process and $\nu_{(CPA-MCL)}$ after the coating process for a deposition



time $t = 4$ min. For this coated PA-MCL, the ratio $\nu^2_{(CPA-MCL)}/\nu_0^2$ is 0.9778. The PE-MCL, simultaneously coated with the PA-MCL, also exhibited a shift in its resonant frequency such that $\nu^2_{(CPE-MCL)}/\nu_0^2 = 0.9864$, as shown on the bottom of Figure 4a. Unlike the two Au-coated MCLs (for which the two ratios were equal: 0.8965 and 0.8967), these two simultaneously Co-coated



MCLs exhibited different mechanical behaviour depending on the position of the cantilever during the coating process; when the MCL was parallel to the cone generatrix, the PA-MCL, the ratio was 0.9778, and when the MCL was perpendicular to the cone generatrix, the PE-MCL, the ratio was 0.9864. This effect remained when

the deposition time increased. Figure 4b shows the results when the two simultaneously coated MCLs were consecutively coated for other 4 min; that is, for a total deposition time of 8 min. Having demonstrated that the amount of material deposited onto each MCL was equal, we can remark that the spring constant of each Co-coated PA- or PE-MCL changed according to the longitudinal or transverse orientation of the film's nano-strings.

Figure 5 shows the changes in the ratio $v^2_{(C-MCL)}/v_o^2$ with consecutive Co deposition times of 15 s. Ratios are displayed for both the PA-MCL and the PE-MCL. These results indicate that there is no difference between the PA- and PE-MCL with regard to these parameters until ≈ 1.0 min and that the same decrease occurs for both with time. The lack of difference may stem from the equal mass deposited on both MCLs and the equal k_0 spring constants for both MCLs. No film was formed, only islands of Co were present and no change of the k_0 of each MCL took place. After percolation, after ≈ 1.2 - 1.4 min of deposition, the slope of the ratio $v^2_{(C-MCL)}/v_o^2$ versus the deposition time, changed. The decrease in $v^2_{(C-MCL)}/v_o^2$ produced by the increase in m was balanced out by the increase in k produced by the percolated film. Because the same quantity of material was deposited on the two simultaneously coated MCLs, the division of the value of $v^2_{(C-MCL)}/v_o^2$ (starting at approximately 2.0 min) must have been a result of the newly generated nano-strings, which produced different values of k for each MCL. In fact, the coated PE-MCL with longitudinal nano-strings exhibited a value of k higher than the corresponding value for the coated PA-MCL with transverse nano-strings.

At higher deposition times, when the nano-strings had begun to grow successfully, the difference between the mechanical behaviour of the simultaneously off-normal coated PA- and PE-MCLs increased, as shown in Figure 6. The changes in the ratio $v^2_{(C-MCL)}/v_o^2$ with a Co consecutive deposition time of 1.0 min (see Figure 6a) show how this ratio for the CPA-MCL (featuring the transverse nano-strings) has a slope practically equal to its initial slope and consistent with the increase in mass of the MCL. The slope for the CPE-MCL (with longitudinal nano-strings) is lower than the slope for the CPA-MCL, and because the increase in mass was equal for both MCLs, an increase in the value of the spring constant, k_0 , must have occurred for the CPE-MCL. One preliminary conclusion can be made: the off-normal Co-coating process increased the spring constant of the MCL with longitudinal nano-strings, whereas for the MCL with transverse nano-strings, which was coated during the same process, only small changes of its spring constant occurred.

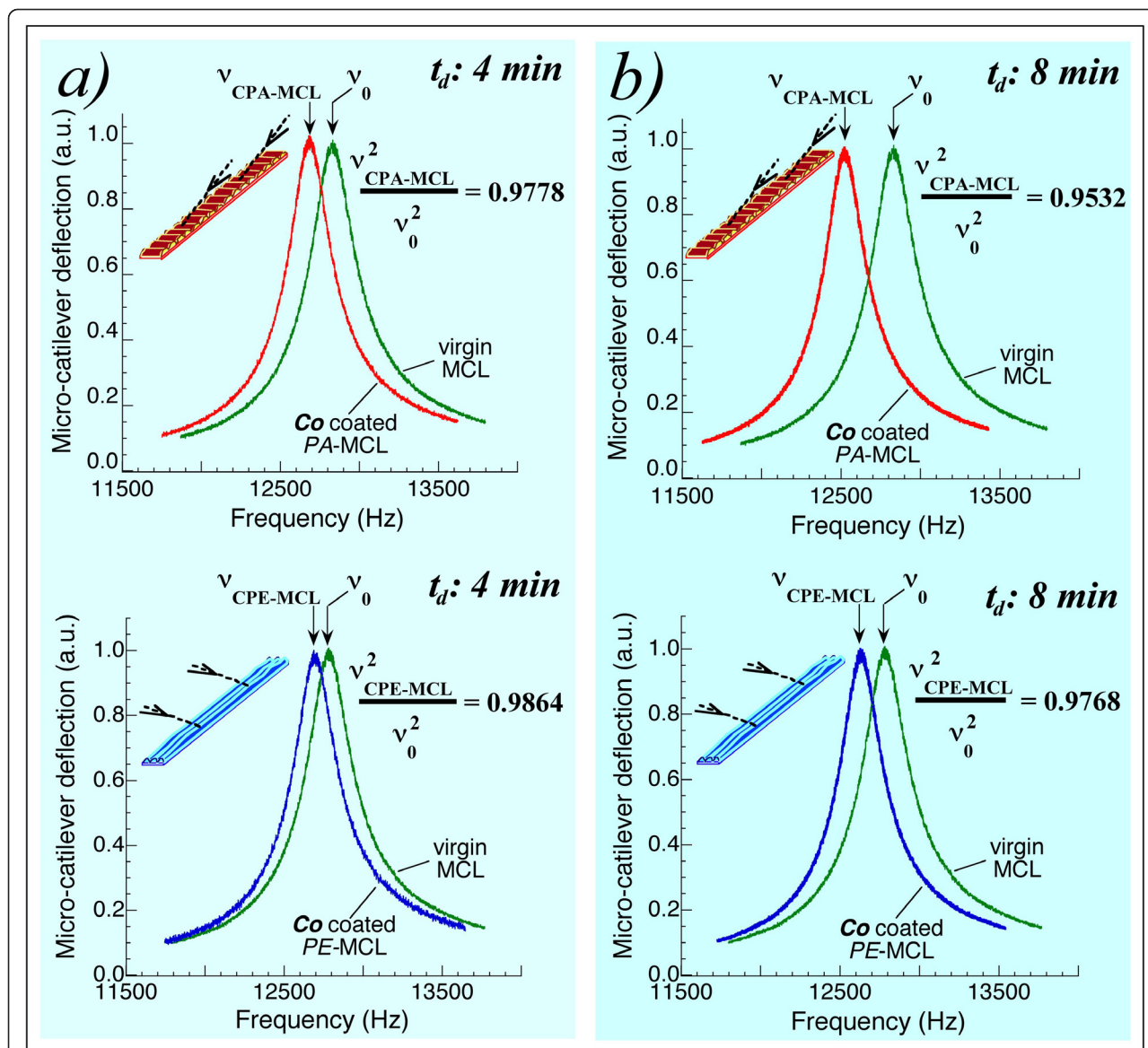


Figure 4 Resonant frequencies of two simultaneously Co coated MCLs: anisotropic elasticity of the films. (a) (Top) Resonant frequencies for the PA-MCL: v_0 representing the resonant frequency before the coating process and $v_{\text{CPA-MCL}}$ representing the resonant frequency after the Co coating process with deposition time $t = 4$ min. $v_{\text{CPA-MCL}}^2/v_0^2 = 0.9778$. (Bottom) Resonant frequencies for the simultaneously coated PE-MCL: v_0 representing the resonant frequency prior to the coating process and $v_{\text{CPE-MCL}}$ representing the resonant frequency after the coating process; $v_{\text{CPE-MCL}}^2/v_0^2 = 0.9864$. (b) Resonant frequencies of the two simultaneously coated MCLs coated consecutively for 4 min: that is, for a total deposition time of 8 min. $v_{\text{CPA-MCL}}^2/v_0^2 = 0.9532$ and $v_{\text{CPE-MCL}}^2/v_0^2 = 0.9768$. Note the significant difference between the PA-MCL and PE-MCL ratios for the two cases and the difference between these results and those displayed in Figure 2.

This behaviour was also observed for other two simultaneously off-normal Co-coated MCLs with a consecutive deposition time of 4.0 min, as shown in Figure 6b.

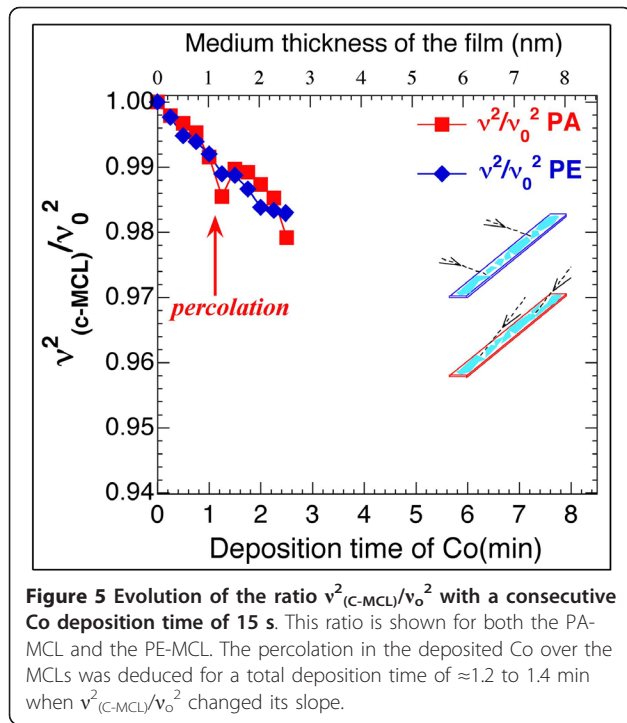
Taking into account [16,17] that

$$v_0 = (C^2 t / 2\pi L^2) (E_0 / 12 \rho_0)^{1/2} = 0.162 (t / L^2) (E_0 / \rho_0)^{1/2} \quad (1)$$

being $C = 1.875$ and the resonant frequencies v_0 (in the interval (8665 ± 5) Hz), the density of the Si ($\rho_0 =$

$2.33 \times 10^3 \text{ kg/m}^3$), the Si Young's modulus ($E_0 = 1.69 \times 10^{11} \text{ Pa}$), and the length ($L = 450 \text{ }\mu\text{m}$) and width ($w = 50 \text{ }\mu\text{m}$) of the two MCLs in Figure 6a, the following values were deduced for MCL: mass, $m_0 = 6.66 \times 10^{-11} \text{ kg}$, $k_0 = 0.200 \text{ N/m}$ and thickness $t = 1.7 \text{ }\mu\text{m}$. The resonant frequency of a coated MCL is [16,17]:

$$v_{\text{C-MCL}}^2 \sim \frac{E_0 (wt/12) + E\delta \{ (w+2\delta) [(1/2) + (\delta/t) + (\delta^2/2t^2)] + t/6 \}}{\rho_0 wt + 2\rho\delta(w+t+2\delta)} \quad (2)$$



where δ is the thickness of the deposited Co film and E its Young's modulus. Considering that $\delta = 10$ nm, that $t \approx 2000$ nm, that $w = 50000$ nm and that the two MCLs are practically equal, we have approximated this last equation, resulting:

$$v^2_{CPE-MCL}/v^2_{CPA-MCL} = (12E_0 + E_{ing})/(12E_0 + E_{trs}) = 1.025, \quad (3)$$

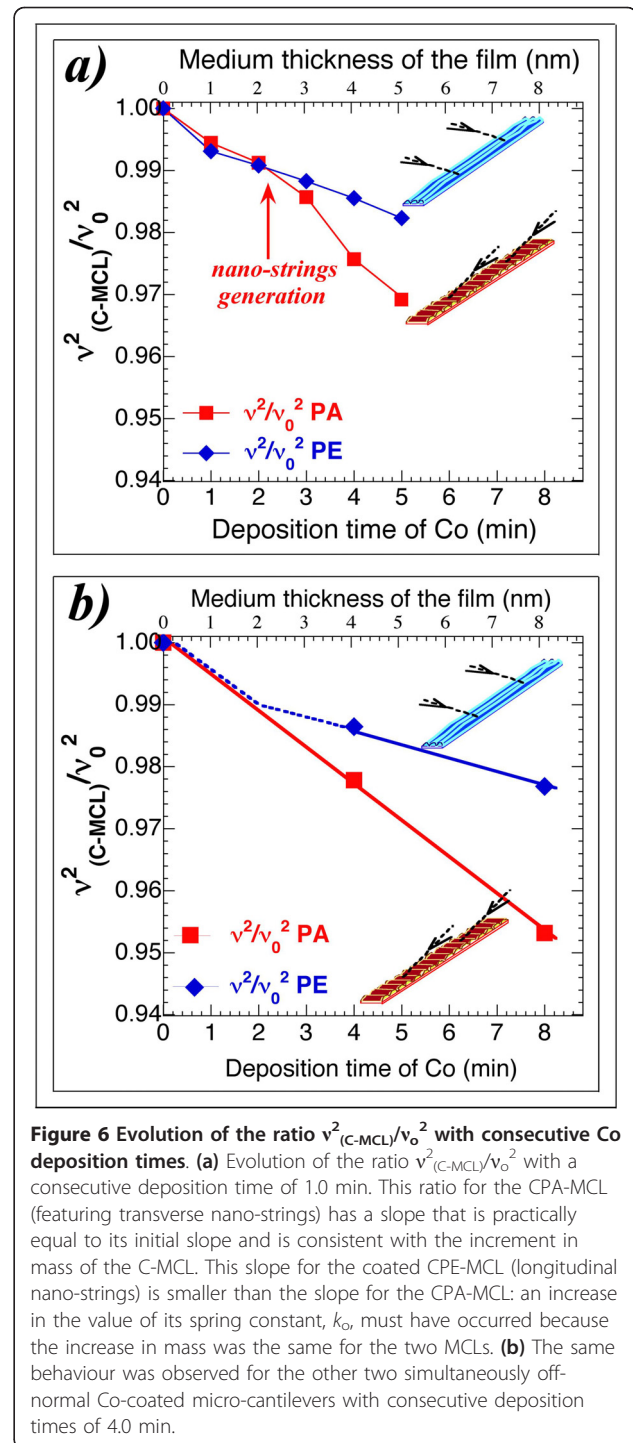
with E_{ing} and E_{trs} the Young's modulus of the CPE film and CPA film, respectively, and 1.025 the experimental value of the MCLs in Figure 4b. Working from this last equation, we obtain the following:

$$E_{ing} - E_{trs} = 0.3E_0. \quad (4)$$

Given the E_0 value, this difference is $\approx 20\%$ of the Young's modulus of the micro-crystalline hcp bulk Co.

Conclusions

A specially designed homemade device combined with a PLD system allowed the off-normal simultaneous coating of two Si MCLs at different controlled locations with respect to the incidence plane of the plasma. For a fixed off-normal angle of $\theta = 55^\circ$, two positions were used for the two MCLs: a position parallel to the incidence plane of the plasma and one perpendicular to that plane. The two off-normal Au-coated MCLs exhibited equal mechanical behaviour, indicating the in-plane isotropic elasticity of these Au pulsed-laser deposited films. This equal mechanical behaviour ensured that the amount of material deposited on both simultaneously coated MCLs



was equal and made it possible to conduct a differential analysis between both. The two simultaneously off-normal Co-coated MCLs exhibited the following behaviour. First, after percolation and nano-string generation, different mechanical behaviour occurred due to the increase in the spring constant for the MCL with Co nano-strings parallel to the longitudinal direction,

whereas the MCL with Co nano-strings transverse to the longitudinal direction experienced changes in the resonant frequency mostly produced by the increase in mass. Secondly, these results were connected with the anisotropic elastic behaviour of the Co film with nano-strings morphology. Thirdly, the Young's modulus of the off-normal deposited Co film was 20% of the Young's modulus of the bulk Co higher for the film direction parallel to the nano-strings than for the film direction transverse to the nano-strings.

Abbreviations

AFM: atomic force microscope; C-MCL: coated micro-cantilever; CPA: film deposited over the microcantilever parallel to the cone generatrix; CPE: film deposited over the microcantilever perpendicular to the cone generatrix; CPA-MCL: coated micro-cantilever parallel to the cone generatrix; CPE-MCL: coated micro-cantilever perpendicular to the cone generatrix; C(PA or PE)-MCL: coated micro-cantilever parallel to the cone generatrix or coated micro-cantilever perpendicular to the cone generatrix; MCLs: micro-cantilevers; Nd:YAG: neodymium-doped yttrium aluminium garnet; PA-MCL: micro-cantilever parallel to the cone generatrix; PE-MCL: micro-cantilever perpendicular to the cone generatrix; PLD: pulsed laser deposition; STM: scanning tunnelling microscopy.

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Authors' contributions

VM, CF and JV participated from the beginning in devising the different steps of the work. Specially, VM with the preparation of the device for off-normal PLD, supports and microcantilever holders and during the coating processes. CF with mechanical characterization of microcantilever and subsequent determination of mechanical resonances. JV with the STM surface observation nano-strings of the microcantilever and VSM magnetic determinations. VM, CF and JV participated at the discussions and analysis of the results and during the preparation of manuscript. Specially CF dedicated extra time for this part.

Competing interests

The authors declare that they have no competing interests.

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