INTERNAL FRICTION AND YOUNG'S MODULUS MEASUREMENTS ON SiO2 AND Ta2O5 FILMS DONE WITH AN ULTRA-HIGH Q SILICON-WAFER SUSPENSION

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INTERNAL FRICTION AND YOUNG'S MODULUS MEASUREMENTS ON SiO_2 AND Ta_2O_5 FILMS DONE WITH AN ULTRA-HIGH Q SILICON-WAFER SUSPENSION

BADANIA TARCIA WEWNĘTRZNEGO I MODUŁU YOUNGA W WARSTWACH SiO_2 I Ta_2O_5 PRZEPROWADZONE W UKŁADZIE ZAWIERAJĄCYM MOCOWANIE PRÓBKI W POSTACI WAFLI KRZEMOWYCH, KTÓRE CHARAKTERYZUJE SIĘ SKRAJNIE WYSOKĄ WARTOŚCIĄ PARAMETRU Q

In order to study the internal friction of thin films a nodal suspension system called GeNS (Gentle Nodal Suspension) has been developed. The key features of this system are: i) the possibility to use substrates easily available like silicon wafers; ii) extremely low excess losses coming from the suspension system which allows to measure Q factors in excess of 2×10^8 on 3" diameter wafers; iii) reproducibility of measurements within few percent on mechanical losses and 0.01% on resonant frequencies; iv) absence of clamping; v) the capability to operate at cryogenic temperatures. Measurements at cryogenic temperatures on SiO_2 and at room temperature only on Ta_2O_5 films deposited on silicon are presented.
Keywords: Ultra low loss suspensions, Q measurements, optical dielectric coatings, silica, tantala Ta_2O_5, thin films

Aby umożliwić prowadzenie badań tarcia wewnętrznego (Q^{-1}) w cienkich warstwach opracowano węzłowy układ zawieszenia o nazwie GeNS (Gentle Nodal Suspension). Do najważniejszych cech charakterystycznych tego układu zaliczamy: i) możliwość wykorzystania łatwo dostępnych substratów, takich jak wafle krzemowe; ii) bardzo niskie straty nadmiarowe pochodzące z samego układu zawieszenia, co umożliwia pomiar wielkości Q przekraczających wartość 2×10^8 na waflach o średnicy 3"; iii) powtarzalność wyników pomiarów strat mechanicznych w zakresie kilku procent, a częstotliwości rezonansowej w zakresie 0,01%; iv) brak mocowań próbki; v) możliwość prowadzenia pomiarów w temperaturach kriogenicznych. W pracy przedstawiono wyniki pomiarów uzyskanych w temperaturach kriogenicznych dla SiO_2 i przy temperaturze pokojowej dla warstw Ta_2O_5 osadzonych na krzemie.

1. Introduction

In recent years an increasing number of experiments and devices have been limited by the mechanical thermal noise of their components: detection of the radiation pressure fluctuation on micro resonators due to the quantum nature of light [1]; sub-Hertz optical resonators used as a frequency standard [2]; detection of Gravitational Waves [3]. Thermal noise arises from the dissipation mechanisms inside the materials that distribute the thermal mean energy all over the frequencies. In a lossless material the same energy is instead concentrated at the resonant modes. The proportionality between power spectral density of thermal noise and internal friction (mechanical loss angle $\tan(\delta)$ or ϕ) is given by the Fluctuation-Dissipation Theorem [4]; for mechanical experiments and the concept of mechanical spectroscopy the work of Saulson [5] and Magalas [6, 7] give a nice introduction to the subject.

Although thermal noise is ubiquitous, the contribution coming from the optical coatings is dominant in all the experiments and detectors mentioned above [8]. The Laboratoire des Matériaux Avancé (LMA), in Lyon, France, is producing the reflective coatings for the main mirrors of the GW detectors Advanced Virgo and Advanced LIGO. These optical coatings are made by stacks of alternate layers of two types of glasses: silica (SiO_2) and titania doped tantala (TiO_2:Ta_2O_5). As compared to metallic coatings this method based on Bragg reflectors assures the lowest optical absorption and making extremely low optical absorption mirrors is part of the know-how that makes LMA a reference laboratory in this field (recently an absorption of 0.14 ppm has been measured on the Advanced LIGO mirrors). In addition, the materials developed at LMA have shown the lowest internal friction value in optical films (doped tantala: $\phi = 2.4\times10^{-4}$; silica: $\phi = 4.6\times10^{-5}$; multilayer high reflectivity: $\phi = 2.8\times10^{-4}$) [9]. In this paper we use the notation ϕ to indicate the $\tan\delta$.

In order to reduce further the thermal noise in coatings LMA and other laboratories in the world have started several R&D programs focused on various aspects of the problem: new materials and alloys [10]; post deposition treatments [11,12]; novel deposition techniques [13]; investigation of the

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structure at the atomic level [12,14]. All these efforts aim to reduce the internal friction by at least a factor 3 (at room temperature and at 10K) with respect the current values of mechanical losses. With this improvement the new optical coatings will be ready for the next generation of GW detectors [15,16].

Coatings are deposited on substrates and their internal friction and Young's modulus are worked out once the double measurements on a naked and a coated sample are compared. The presence of a substrate introduces a large background and reduces the sensitivity of the measurements to the coating parameters, a phenomenon that can be called *dilution*. In detail, using the definition of quality factor $Q=1/\phi$, the relation between the three loss angles of the composite resonator (substrate + coating) ϕ_{res}, of the coating ϕ_{coat} and of the substrate ϕ_{sub}, reads:

$$\phi_{\mathrm{res}} = \frac{2\pi \mathrm{E_{coat}}\,\phi_{\mathrm{coat}} + 2\pi \mathrm{E_{sub}}\,\phi_{\mathrm{sub}}}{2\pi \mathrm{E_{res}}} = D\,\phi_{\mathrm{coat}} + (1-D)\;\phi_{\mathrm{sub}}, \tag{1}$$

where E_{coat} and E_{sub} are respectively the elastic energies stored in the coating and in the substrate. The sum of them gives the total energy of the resonator E_{res}. *D*, the *dilution factor*, is then defined as the ratio E_{coat} / E_{res}. Equation (1) is used here to work out the coating loss angle from the double measurement of ϕ_{res} and ϕ_{sub}. The dilution factor *D* can be either calculated through the use of Finite Element Analysis (FEA) software or indirectly measured through the frequency shift of the resonator caused by the deposition of the coating (resonant method). The relation that has been used is:

$$D = 1 - \left(\frac{\mathrm{f^u}}{\mathrm{f^c}}\right)^2 \frac{\mu_{\mathrm{sub}}}{\mu_{\mathrm{coat}} + \mu_{\mathrm{sub}}}, \tag{2}$$

where $f^{\,u}$ and $f^{\,c}$ are respectively the resonant frequencies of the uncoated and coated resonator; μ is the surface mass density (kg/m^2), the ratio of surface mass densities can be replaced by the ratio of masses: m_{sub}/m_{res} because the surface is the same for coating and substrate. Details on the derivation of Eq. (2) can be found in Ref. [17].

It can be shown that the dilution factor is proportional to the coating Young's modulus. In fact, in the case one deposits a coating with zero Young's modulus, the energy stored in the coating is zero too. To check the consistency of Eq. (2), the frequency ratio squared in this case is exactly equal to the mass ratio m_{res}/m_{sub} (frequency is reduced by the effect of inertia increment), which makes *D*, again, null. Comparing the dilution factor *D* measured by the frequency shift with the one coming from the FEA software we are able to measure the coating Young's modulus using that of the substrate as a reference.

Due to the low loss materials tested and the effect of dilution other measuring methods like an inverted torsion pendulum and dynamic mechanical analyzer (DMA) cannot be used in this type of research.

2. The gentle nodal suspension

The nodal suspension proposed here (GeNS) has been developed originally at the INFN laboratories in Firenze, Italy [18]. It consists of suspending the plate resonator of thickness t on top of a spherical surface of radius R. As long as t <2R and there is no sliding between the contacting surfaces, the plate sits in a stable equilibrium. Details of the system can be found elsewhere [18]; here we report on GeNS operated at cryogenic temperature. A picture of the system is shown in Fig. 1 where the side view of a 3" silicon wafer is suspended over a plano-convex silicon lens of 63 mm radius of curvature. The lens support is made of copper. The sample is excited by an electrostatic actuator and the vibration is detected by the laser beam deflection method using a pair of photodiodes placed at the end of an optical lever system. Several modes, up to 4, have been excited consecutively and their signals have been amplified, acquired and processed at the same time by a Labview®[19] based code. After the sampling (at a frequency ranging from 10 kHz to 50 kHz) the signal is digitally converted (16 bits ADC) and Fast Fourier Transformed.

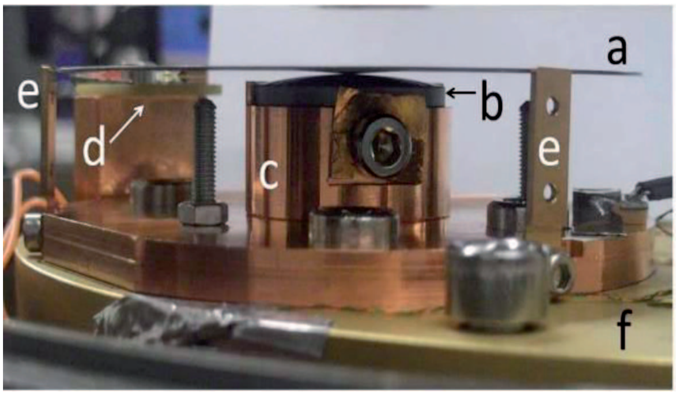


Fig. 1. The GeNS suspension mounted in the cryostat at LMA, side view: a) 3" silicon wafer; b) silicon lens; c) copper support for the lens; d) electrostatic excitation placed below the wafer; e) safety stands; f) cryostat cold plate. The temperature is measured on the cold plate. The laser comes from the top and senses the vibration on a point close to the disc edge

The amplitude spectral density of the photodiodes signal is calculated by FFT and then divided into frequency bands of 5 Hz widths, centered on each excited mode n. The maximum amplitude inside each frequency interval is recorded and this constitute the temporal narrowband signal $A_n(t)$. Care has been taken to assure that the frequency bins are much larger than $1/\tau_n$, where τ_n is the characteristic decay time of the mode n. In order to measure τ_n a linear fit of the data $\ln[A_n(t)]$ vs. t is done [20, 21, 22]:

$$ln\;[A_n\,(t)] = -\frac{t}{\tau_n} + ln\left[A_n^0\right]. \tag{3}$$

The quality factor of the mode is then calculated through the relation $Q = \pi\, f_n \tau_n$, where f_n is the mode frequency. Using 460 μm thick silicon [100] wafers the specific dilution factor D_S is about 4.7×10^{-3} 1/(μm 100GPa) (i.e. per μm of coating with Young's modulus 100 GPa and deposited on one side).

3. Measurements

In this paper two measurements are presented. The first regards the loss of deposited silica, measured from 14K to

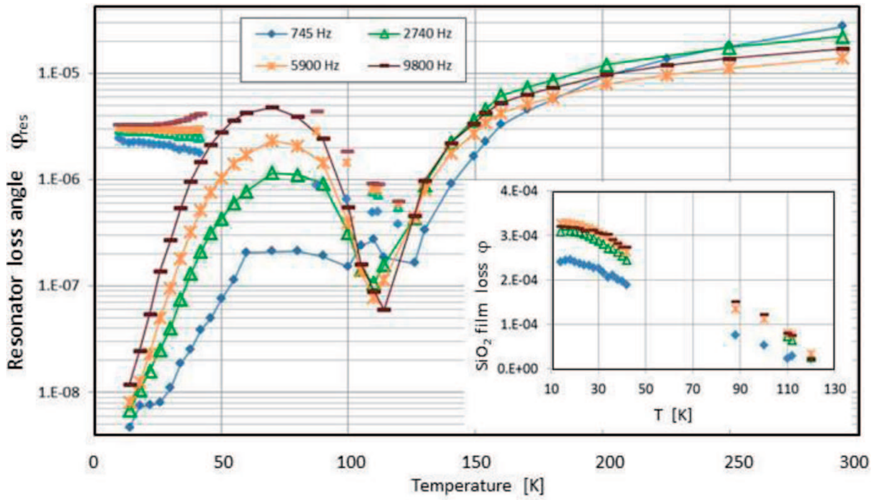


Fig. 2. Internal friction measurements on a 3" silicon wafer (points connected by a line) and on a wafer coated with 2.5 μm of SiO_2 (isolated points). For clarity only 4 modes out of the 9 measured in total are represented. For the coated sample there is a gap between 42K and 88K due to problems with the cryostat. The highest measured Q = 2.15×10^8 was taken at 14K for the mode 745 Hz. Except below 25K at all temperatures the thermoelastic loss dominates. The peak around 70K is due to a minimum in the thermal expansion of silicon. Bellow of the 120K down to 18K the Coefficient of Thermal Expansion is negative. Around the 120K, CTE is zero for the silicon and the loss of all the modes converges to almost the same value. Inset: loss angle of the SiO_2 film following Eq. (1) using the dilution *D* as calculated by FEA (the average value is about 1/114). The SiO_2 coating was annealed at 500°C after deposition

120K; the results are presented in Fig. 2. The second measurements regard deposited tantala (Ta_2O_5), shown in Fig. 3 c). In Fig. 3 a) the effect of the curvature, induced by the stress in a silica film, on the resonant frequency is presented. Because of this effect the measurement of Young's modulus through frequency shift can be done only if the film is deposited on the two sides of the substrate (see Fig. 3 b) and that has been done for the three tantala samples. Two of these samples have been annealed at 400°C and 500°C. Measurements of loss angle of all three Ta_2O_5 samples for different modes are shown in Fig. 3c.

The relation used to calculate the dilution factor, *D*, for a double coating is:

$$D = 2\frac{3\, t_{\text{coat}}\, Y_{\text{coat}}\, (1-\upsilon_{\text{sub}})}{t_{\text{sub}}\, Y_{\text{sub}}\, (1-\upsilon_{\text{coat}})}, \tag{4}$$

where t, Y and υ are respectively the thickness, Young's modulus and Poisson's ratio. The values used in this work are collected in Table 1.

TABLE 1

Parameters used in Eq. (4) and Fig. 3 b)

	t [μm]	Y [GPa]	ν
coat – Ta_2O_5	2.38	140*	0.23*
sub – Silicon [100]	467	130	0.277

*These values have been taken from Ref. [23].

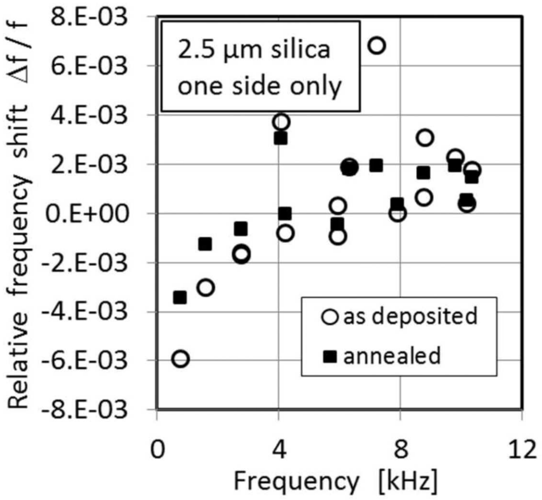
4. Comments and conclusions

Measurements of Ion Beam Sputtering (IBS) deposited silica shown in Fig. 2 can be compared with recent measurements done by other colleagues [25] on a material deposited on silicon cantilevers by the same technique. The cantilevers are clamped on a thicker end. There are several differences between the two results: i) our results do not show any loss peak at all the frequencies investigated whereas [25] reports a peak around 20 K that follows the Arrhenius' law; ii) in our results the loss increases with frequency whereas in [25] the loss has a minimum at about 7.3 kHz; iii) the loss in our material seems to be about a factor 2 lower than that of [25]. These differences may be explained by a different choice of deposition parameters and by some effect due to the cantilever clamp which is absent in our system.

Fig. 3 a) shows the effect of curvature on the resonant frequency of the silicon disc. Beside the disagreement between our measurement and the simulation reported in [24] as explained in the caption, it is not clear yet if the curvature has an effect on the loss measurements. Investigations are on-going on this issue.

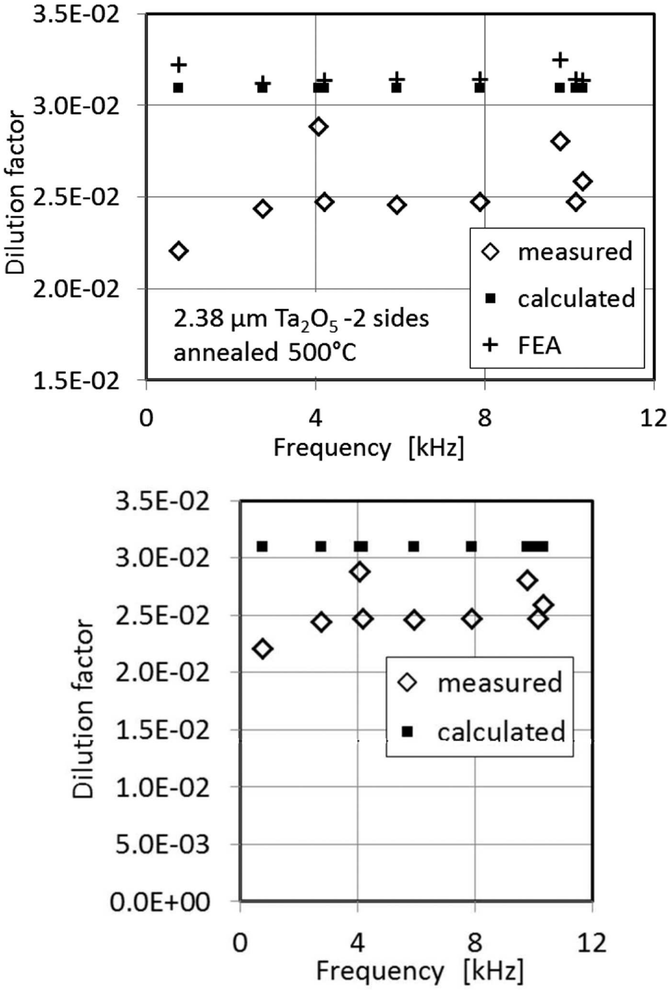
The measurement of dilution factor proposed here allows determine the film loss angle without the knowledge of the Young's modulus and thickness of substrate and film. It is sufficient to measure the resonant frequency and the mass of the resonator before and after the coating deposition, as indicated by Eq. (1) and (2). This method relies on the reproducibility on the measurement of resonant frequencies: with our system GeNS we have a fractional reproducibility of 10^{-4}. On the contrary, in order to measure the film Young's modulus with respect to that of the substrate one needs the measurement of substrate and film thicknesses.

From the difference between the calculated and measured dilution factor (see Fig. 3 b and Eq. (4)), a tantala Young's modulus of about 110 GPa has been worked out. The difference may be explained by a different choice of deposition parameters between our samples and those of reference [23] from where the tantala Young's modulus used in the calculations and FEA [17] has been taken.



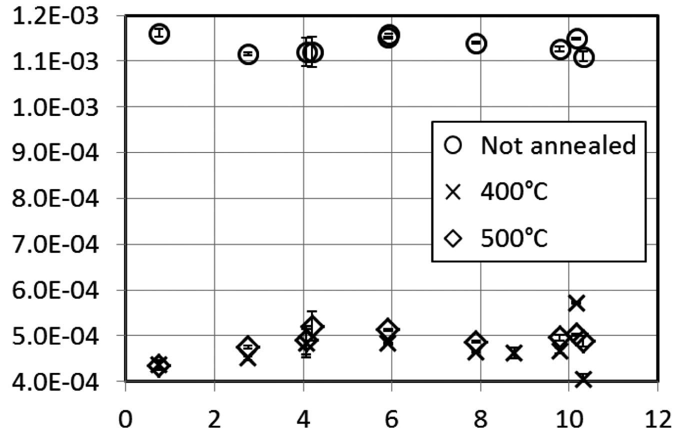
(a)

Fig. 3. a) Effect of the curvature on the frequency shift Δf of each mode: the as-deposited silica has a compressive stress of about 200 MPa; after annealing at 500°C the stress is reduced. On a flat plate the frequency shift is mode independent. The effect of biaxial curvature reduces the resonant frequencies and this is in contradiction with what is presented in the work of Lauwagie [24] where the curvature in rectangular plates always increases the frequencies



(b)

Fig. 3. b) Comparison between the dilution factor measured by frequency shift through Eq. (2); that calculated by Eq. (4) and the one estimated by the definition Eq. (1) with the aid of a FEA code. The double side coating shows a much more regular frequency shift and dilution factor than the disc coted on one side only as shown in Fig. 3 a. The calculated and FEA data points assume a tantala Young's modulus of 140 GPa. The difference between measured and calculated dilution factor has been attributed to the film Young's modulus



(c)

Fig. 3. c) Measurements of Ta_2O_5 loss angle for different modes: the three sets of data correspond to as-deposited, annealed at 400°C and annealed at 500°C samples. The last two are equal within the experimental errors. The measured dilution factor has been used in all the three sets of data. The error bars have been estimated from three sets of measurements performed on a naked disc on three different days. For the uncertainty estimation the maximum variation of measured loss has been considered here

Finally Fig. 3 c) shows the measurement of loss angle on as-coated and annealed tantala. In order to estimate the uncertainties an uncoated disc has been measured three times taking out and replacing it back on GeNS. The maximum variation of loss angle varies from mode to mode and it is in the range $2.5{\times}10^{-8}$ (mode at 5.9 kHz) to $8.4{\times}10^{-7}$ (mode at 4.2 kHz). The uncertainties on the mechanical loss of coatings reported as error bars in Fig. 3 c), have been calculated from these values considering Eq. (1). The variation of loss observed from mode to mode is larger than the statistical uncertainty. On the contrary, from other measurements [17], we know that the expected loss of sputtered tantala is constant with frequency: that means other systematic effects related to the presence of coatings have to be considered in order to justify the observed variation of loss. The loss decreases with the increase of annealing temperature until the material becomes polycrystalline above 600°[26].

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