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E Cesarini, M Prato, M Lorenzini, G Cagnoli, E Campagna, et al.. Mechanical characterization of uncoated and Ta₂O₅-single-layer-coated SiO₂ substrates: results from GeNS suspension, and the CoaCh project. *Classical and Quantum Gravity*, 2010, 27 (8), pp.84031. 10.1088/0264-9381/27/8/084031 . hal-00587622

HAL Id: hal-00587622

<https://hal.science/hal-00587622>

Submitted on 21 Apr 2011

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Mechanical characterization of “uncoated” and “Ta₂O₅-single-layer-coated” SiO₂ substrates: results from GeNS suspension, and the CoaCh project‡

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Abstract. Thermal noise of the mirrors limits the sensitivity of interferometric gravitational wave detectors in the frequency range between 50 to 300 Hz. According to the Fluctuation-Dissipation theorem, the thermal noise amplitude is proportional to the mechanical loss of the mirrors. Fused silica substrates loss angle widely ranges from 10^{-9} to 10^{-6} , while for the coating it is in around 10^{-4} . Loss angle measurements in ultra low mechanical loss materials are normally affected by a large systematic error due to the excess losses introduced by the suspension system used to hold the samples. An innovative measurement system (**GeNS, Gentle Nodal Suspension**), where the disk-shaped sample is suspended in equilibrium on the top of a sphere, touching in one of the nodal points of vibration, has been developed in INFN Florence Virgo laboratory. The advantages of this system are: i) the good reproducibility of loss angle measurements; ii) one surface only (in any case uncoated) of the sample is touched; iii) the contact surface is minimized because of the absence of applied forces. This suspension has been used to characterize annealing and coating deposition effects on mechanical quality factor Q . An interesting comparison/analysis of these effects on mechanical, optical, chemical and surface properties using spectroscopic ellipsometry, X-ray photoelectron spectroscopy and atomic force microscope has been carried out.

PACS numbers: 04.80.Nn, 06.60.Ei, 62.40.+i, 68.37.-d

‡ This work has been partially supported by the Italian Ministry of University, Education and Research-MIUR under the Contract PRIN -2007T7AC3L

1. Introduction

The sensitivity of an interferometric 'gravitational waves (GW)' detector is primarily limited by the degree to which the test masses of the detector can be isolated from sources of displacement noise. Thermal excitation of the degrees of freedom of the suspended masses results in a displacement noise at the output of the detector. The thermal noise is indeed the limiting noise in the region from 50 to 200 Hz for the next generation of optical ground-based GW interferometric antennas [1].

According to the fluctuation-dissipation theorem, thermally activated equilibrium fluctuations are determined by the mechanical dissipative characteristics [2]. While in the present day detectors the limiting factor is the bulk thermal noise, in second generation detectors new substrates, with a different volume to surface ratio and lower losses, will be employed and dissipative processes in the thin multilayered reflective coating, deposited on the mirrors, will be dominant [3][4].

A big effort is being carried on to select the best materials for realizing mirror substrates with very low mechanical dissipation and to understand the loss mechanisms affecting the coatings, hopefully finding the way to produce films with a very low loss angle φ , or equivalently with a very high mechanical quality factor Q . For this purpose it is mandatory to determine in a reliable way the loss angle of mirrors or of samples made of the same material. Moreover a systematic study of materials, deposition and processing technique is therefore required to optimize the mirror characteristics in order to improve the sensitivity without spoiling the optical performances.

In this paper a very short review of the experimental methods used to measure the mechanical performances of a mirror substrate is presented (this Section) and the innovative suspension method used to hold the sample during the measurement is described, together with the results obtained by this new set up (Section 2). The mechanical measurements have been compared after with surface analysis results trying to find a possible correlation (Section 3). This kind of approach will be used also to investigate dissipative mechanisms in multilayered coated samples: the general guidelines of this project are shortly outlined.

1.1. Loss angle measurements

The loss angle φ can be measured through the "ring-down" technique, where the damping of a free oscillation is recorded. If the oscillation is at the normal frequency ω_0 , the damping envelope is an exponential decay whose rate is proportional to the loss angle φ at that frequency: $\varphi(\omega_0) = 1/Q = 2/(\tau\omega_0)$, where τ is the decay time and Q is the quality factor.

1.2. Issues of suspensions

The Q measurements entail the sample to be held by some suspension or clamping where the energy transferred from the sample to the suspension is negligible. This point

is not trivial because the energy loss by suspension or clamping may affect the results with unknown systematic error. To face this problem several solutions were adopted by different groups working in this field, according to different shapes and dimensions of the samples.

When dealing with thin slabs a massive clamp is usually used to hold the sample [5], while thick cylinders can be suspended using a single wire. The wire is passed around the lateral surface of the cylinder forming a “U-shaped” suspension, narrower in the upper part [6].

If the material under test is fused silica it is possible to weld a thin silica fiber directly on the edge of the sample. The decoupling between the sample and the environment is provided by suitable silica bobs placed along the length of the suspension fiber. In this way a monolithic welded suspension is realized [7].

Cylindrical samples can also be clamped between two spheres, pressing against the center of the two faces: if this is a nodal point for the excited oscillation, no significant energy transfer is expected (for a general approach to nodal suspension see [8])

In all these systems there is no certainty in whether the measured Q is a property related to the sample under investigation or it is significantly determined by the excess loss coming from their setup, especially for sample with high Q in the range of $10^6 - 10^9$. Parameters such as the effective contact surface, the specific damping due to the contact between two materials, and even the precise location of the contact surface, are difficult to quantify and to control.

2. GeNS: an innovative nodal suspension

In order to overcome the problems and the limitations presented by above setups, especially for thin coated samples, we developed a new kind of nodal suspension (**G**entle **N**odal **S**uspension) [9]. In this new solution the sample, which for our purposes can be supposed disk shaped, is suspended in equilibrium on top of a sphere or half-sphere. In this kind of suspension the contact surface is minimized because of the absence of applied forces. It is suitable for studying the mechanical dissipation of coated mirrors too, because it leaves one surface untouched and does not damage the film on the other side. Moreover it allows us to change one at a time the different relevant parameters of the set up that may affect the measurements, in principle to distinguish the intrinsic dissipation of the material from the losses due to the experimental setup.

If ϑ is the angular position of the disk, suspended on the sphere, with respect to the horizontal plane, a stable equilibrium is achieved whenever $D > t$, where D is the diameter of the sphere and t is the thickness of the suspended cylindrical sample. We supposed to be in a pure rolling condition that is, the static friction coefficient μ_s is assumed high enough to avoid any slip during the disk oscillation around the equilibrium position. The angular range where stable oscillation are possible is: $\vartheta \sim \pm \sqrt{3(D - t)}/t$. Usually the stability region is large enough to guarantee a fast and handy manual positioning.

2.1. Results

The GeNS experimental set up is fully described elsewhere [9] (figure 1). During the characterization stage, the measurements of a Suprasil311[®] [10] annealed disk of a diameter of 75 mm diameter and a thickness of 3 mm gave a loss angle φ of $5 \cdot 10^{-8}$. The new suspension does not present any intrinsic limitation to high Q measurements when comparing the results with the ones from other laboratories that measured similar annealed silica samples with the same aspect ratio [11].

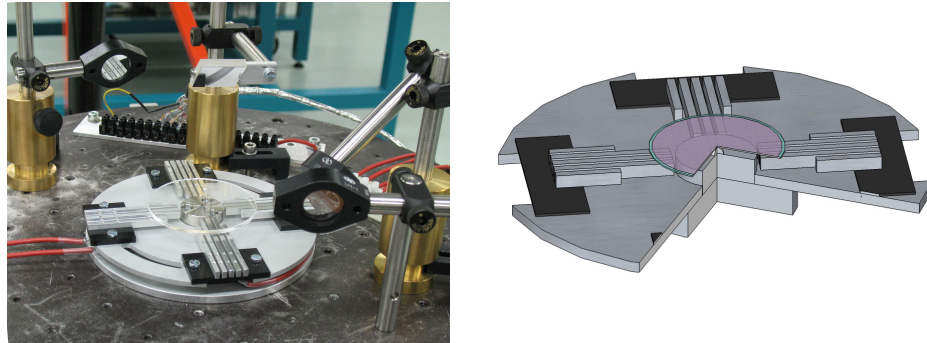


Figure 1. (Left figure) Picture of the fused silica disk on top of GeNS and the mirrors used for the optical lever. (Right figure) A scheme of a section of suspension and excitor system.

Moreover, the authors have shown that the mechanical Q, measured within the same suspension, does not depend on the position of the suspension point: the results are fully reliable even when the sample is not centered on the sphere within an area that is about 3 times bigger than the contact area [9].

2.2. Variability of Q measurements

Even if most of the relevant parameters can be controlled with this setup, still a variability in the Q measured between different suspensions was observed (up to 20%) (figure 2). There was a doubt that this variability can be due to the cleaning of the sample because dust grains were very often identified when a low Q value was obtained. Thus, the facility has been moved in a clean room (ISO7) to investigate the influence of dust deposition on reproducibility. The new measurements inside the clean chamber have shown that the mean Q value is increased, but the variability of the Q value between two different suspensions is still high (figure 2). These measurements suggest that clean chamber operations, while leaving the variability unchanged, allow higher Q values to be achieved. Even though the causes of this dispersion are still not well known and have to be investigated further, it is clearly advantageous to maintain the set up in a clean environment.

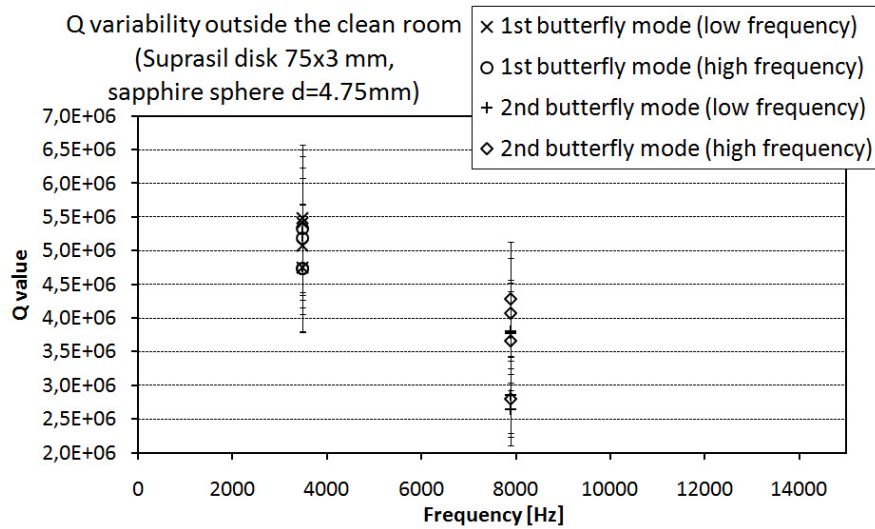


Figure 2. Q vs frequency for a Suprasil311[®] disk with diameter $D = 75$ mm and thickness $t = 3$ mm on a sapphire sphere of diameter $d = 4.75$ mm before the movement in a clean room.

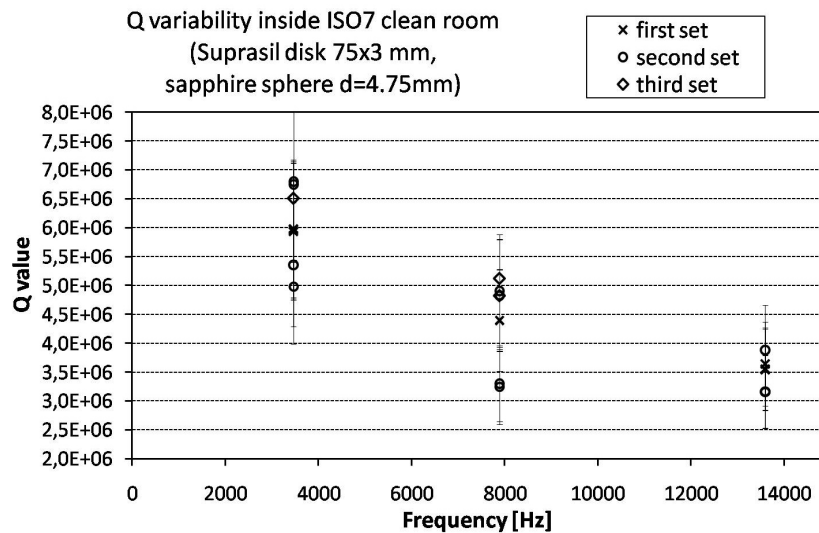


Figure 3. Q vs frequency of the same sample and sphere of the left figure. The mean value for the first butterfly mode is increased from $5.1 \cdot 10^6$ to $6.1 \cdot 10^6$

3. Correlation between Q measurements and surface analyses

Lattice defects and impurities are not distributed homogeneously in the volume of a body. The surface region is constantly exposed to chemical or mechanical damage and it is reasonable that here the amount of defects is much greater than in the volume, so most of the dissipation occurs in the external shell. To investigate how the thermo-mechanical and optical properties are related to the surface quality, a series of surface analyses has been performed on fused silica samples. The same samples have been observed with

different technologies after some treatments, that modified their mechanical quality factor. Here we report the analyses results of the annealing and of Ta₂O₅ monolayer deposition effects.

3.1. Surface Analyses

The analyses performed on fused silica substrates are the Atomic Force Microscope (AFM), the Spectroscopic Ellipsometry (SE) and X-ray Photoelectron Spectroscopy (XPS). This approach aims to understand the extent of surface losses with respect to the bulk ones and their main sources (chemical contamination, mechanical processing, level of roughness, aging and stoichiometry). All the analyses have been performed in correlation with experimental Q value measurements.

Spectroscopic Ellipsometry (SE) SE is a optical technique for the investigation of the dielectric properties of surface or thin films. Standard SE measurements were performed on a rotating compensator ellipsometer (M-2000, J.A. Woollam Co. Inc.). Principles of SE are described at length in literature [12] [13]. The output of ellipsometry is the complex reflection coefficient: $\rho = r_p/r_s = \tan \Psi \exp(i\Delta)$, where r_p and r_s are the Fresnel reflection coefficients for p- and s-polarization, respectively.

Atomic Force Microscope (AFM) The AFM is a very high resolution type of scanning probe microscope, with demonstrated resolution of fraction of a nanometer. Tapping mode AFM measurements were performed using a Multimode/Nanoscope IV system (Digital Instruments) and Si cantilevers (OMCL-AC160 TS, Olympus).

X-Ray Photoelectron Spectroscopy (XPS) XPS is a quantitative technique that measure the elemental composition, empirical formula, chemical state and electronic state of the elements that exist within a material. XPS analysis was carried out with a PHI ESCA 5600 MultiTechnique apparatus. The system consists of an X-ray Al-monocromatised source ($h\nu = 1486.6$ eV) and a spherical capacitor electron energy analyser, used at a constant pass energy of 5.85 eV. In the standard configuration the analyser axis formed an angle (take-off angle) of 68° with the sample surface. The binding energy (BE) scale was referenced to the adventitious C 1s level at 284.8 eV.

3.2. Study of the annealing effects

In the annealing process the glass is heated until the temperature reaches a stress-relief point, that is the annealing temperature (T_A) at which the glass is still hard to deform, but is soft enough for the stresses to relax. The piece is then left into a heat-bath until thermal equilibrium is reached everywhere in the sample [14][15][16]. The time necessary for this step varies depending on the type of glass and its maximum thickness. The glass is then slowly cooled at a fixed rate until its temperature goes below the strain point (T_S). Then the sample can be safely brought to room temperature. The typical

Table 1. Characteristic temperatures used in the annealing process with 2 different fused silica disk samples

Temperature[°C]	HerasilHOQ300 [®] <i>S/V</i> ~ 2.08mm ⁻¹	Suprasil311 [®] <i>S/V</i> ~ 0.72mm ⁻¹
T_A	1180	1120
T_S	1080	1025

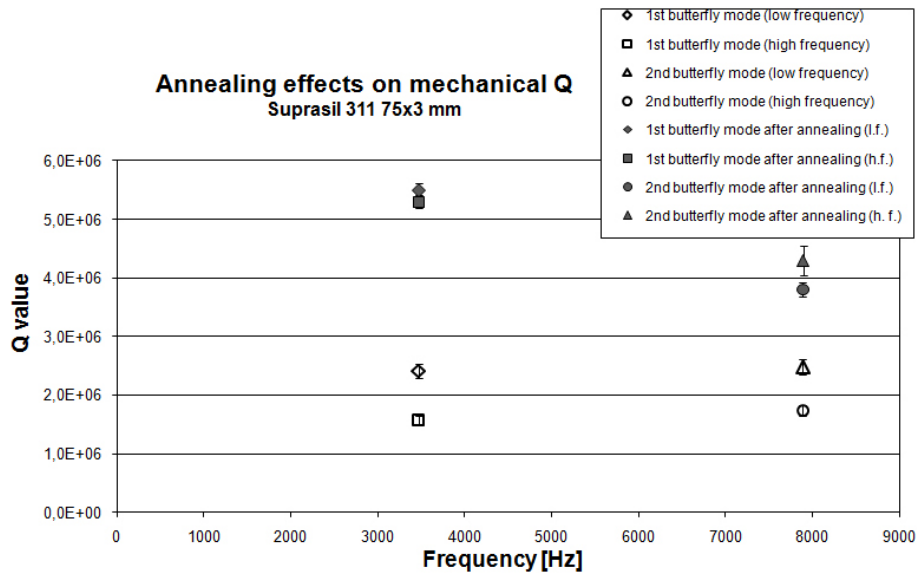


Figure 4. Q vs frequency before and after the annealing treatment at 1120 °C for the Suprasil311[®] disk sample on the sapphire sphere. The maximum Q value of the disk has changed by a factor 1.6 and the values measured for the 2 peaks of the same resonance frequency are closer

temperatures for an HerasilHOQ300[®] [10] disk and a Suprasil311[®] disk with a surface to volume ratio respectively of 2.08mm⁻¹ and 0.72mm⁻¹ were kindly suggested by S. D. Penn (private communication) (Table 1).

The following heat treatments were done in a vacuum tube furnace with a maximum reachable temperature of 1200 °C. To make rough vacuum level (about 10⁻² mbar), a rotary pump has been joined. After the Suprasil311[®] sample annealing the maximum Q value of the disk is improved by a factor 1.6, changing from 3.4 · 10⁶ to 5.5 · 10⁶, and the two peaks of the chosen resonance frequency are closer (figure 4). After the annealing, the maximum Q value of the HerasilHOQ300[®] sample is improved by a factor 1.5, changing from 1.2 · 10⁶ to 1.8 · 10⁶.

Surface roughness, optical properties and chemical contaminations were investigated using AFM, SE and XPS.

AFM images obtained on as received Suprasil311[®] samples (panel a of figure 5) show areas with protrusions, tens of nanometers high, sitting on very flat regions (several μm² wide, RMS roughness of the order of 1 nm). These protrusions (likely fine particles residuals of the polishing process) increase the local RMS roughness up to several

nanometers. After the annealing process, the RMS roughness decreases to less than 1 nm (panels b of figure 5).

The analysis of SE spectra taken on as received samples indicates a roughness of $r_{before} = (0.54 \pm 0.02)$ nm (see [17] for explanation of the fitting procedure). While these particles mainly act as light scattering and depolarizing centers thus affecting the reflectivity of the sample, they have a limited impact on the ellipsometric parameters. Anyway after the annealing process the SE data indicate a surface with a virtually null roughness, in agreement with the AFM observations. This is shown in figure 6 where it can be observed that, after the thermal treatment, the parameter Δ passes from a value of about 179.5 degrees to 180 degrees over the whole spectral range, i.e. the expected value for an ideally flat air/dielectric interface.

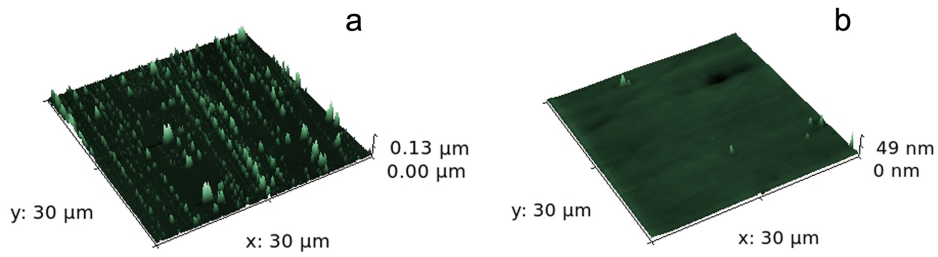


Figure 5. AFM investigation of the effects of the annealing process. Panel a: 3D AFM image of a ($30\mu\text{m}\times 30\mu\text{m}$) area in the center of a Suprasil311[®] sample before annealing. The RMS roughness value is increased by the presence of protrusions, tens of nanometers high. Panel b: 3D AFM image of a ($30\mu\text{m}\times 30\mu\text{m}$) area in the center of a Suprasil311[®] sample after annealing. RMS roughness is now less than 1 nm.

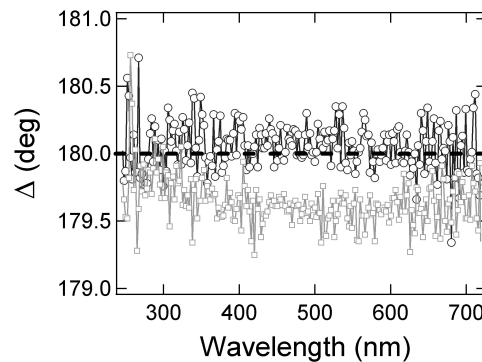


Figure 6. SE Δ parameter before (light gray squares) and after (dark gray circles) the annealing treatment. The ellipsometric surface roughness changed from ≈ 0.54 nm to virtually zero.

The XPS analysis on HerasilHOQ300[®] sample, after the heat treatment, shows that a contamination of the outer layer of the sample by carbon, bonded with the

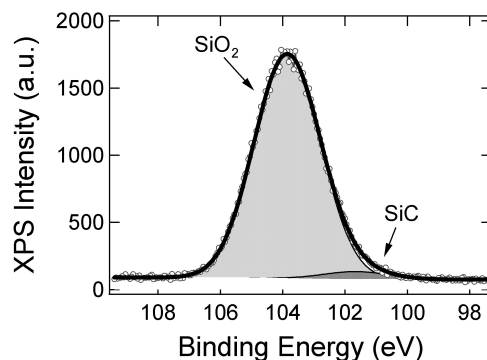


Figure 7. XPS surface chemical analysis. High resolution image of the Silicon peak of the HerasilHOQ300[®] disk after the annealing treatment. A 8% peak of silicon carbide is present.

silicon, has been occurred. In a high resolution image of the silicon energy peak after the treatment, a small peak (about 8% of the total area) at 101.4 eV has been observed. This energy range is typical of the silicon carbide (SiC) (figure 7). Probably the carbon already present in the furnace and upon the surface of the disk has been bonded to silicon due to the high temperature reached during the thermal treatment. This results suggest that a higher level of vacuum or a controlled atmosphere with a continuous flux of Argon is necessary, during the annealing process.

3.3. Study of the coating deposition

To investigate what phenomenological parameters, modified during a controlled treatment, are important for mechanical dissipation, can be a useful mean for trying to characterize and model coating dissipation. Without modifying in principle the optical configuration of the interferometers, several attempts have been made for improving the global (substrate and coating) quality factor of the mirrors. A good approach is to optimize the dimensions of each layer [18]; this approach may be considered not alternative but complementary to other approaches aiming to improve the performances of the layers by modifying their structure and/or composition. In this sense experiments have been performed introducing a dopant in the Tantalum Oxide, leading to interesting results [19] [20]. Following the line of modifying the structure of the layers, either internal or at the interface surface between layers, we propose to perform systematically a set of different measurements on many samples in order to give an almost complete characterization of them, followed by realistic modeling: this process will be iterated in order to introduce step by step the suitable modifications in the samples until a stable “optimum” performance is obtained both from optical and thermo-mechanical point of view. This project about coating characterization has been funded by the Italian Ministry of Research and Education. Independent measurements of phenomenological parameters (optical properties, chemical properties and structural properties) and loss

angles in different controlled experimental conditions may help in defining a strategy to modified related structures of the internal friction spectrum.

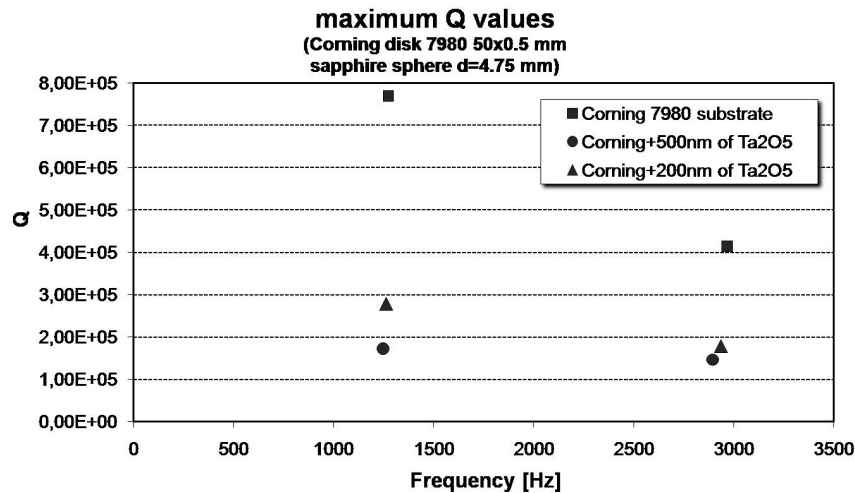


Figure 8. Q vs frequency of Corning7980[®] disk substrate and Corning7980[®] disk substrates with two different deposited monolayer of Ta₂O₅ (200 nm and 400 nm).

The starting point has been the acquisition of a set of equal samples of single Ta₂O₅ layer on SiO₂ substrate (Corning7980[®], diameter of 50 mm, thickness of 0.5 mm). We determined for the blank substrate the Q value. Then we measured the same parameter after the deposition of the single Ta₂O₅ layer (200 nm and 500 nm) (figure 8). The coating deposition has been provided by ATFilms with a double ion beam sputtering technology. These data are only preliminary measurements to check the reliability of the set up for the purpose of the CoaCh project. More measurements will be done to increase the statistics and better samples (with higher quality fused-silica and with Virgo-like coating) will be employed. First surface analyses on coated samples are reported elsewhere [17].

4. Conclusions

The GeNS suspension has been used to perform mechanical Q measurements. This set up has still a residual variability in the measured Q among two different suspensions, at the level of what is found by other groups with different set up. Anyway, considering the possibility of an easy control of the parameters and the fact that this suspension allow not to damage the coated face of the sample, it has been employed to study the annealing and coating deposition effects on Q measurements.

Trying to correlate the change in mechanical performances with optical, chemical, structural and tribological surface properties, some surface analyses have been performed before and after the annealing treatment. The optical properties and the surface roughness improve with the increment of the mechanical Q. Contamination of the outer

layer of the sample by carbon, bounded with the sample silicon has been observed. This is probably due to the rough level of vacuum during the heat treatment.

The same approach will be used to investigate what phenomenological parameters, modified during a controlled treatment, are important for mechanical dissipation in coated sample, in order to obtain a stable “optimum” performance of the coating layer.

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