Low temperature mechanical dissipation of an ion-beam sputtered silica film

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Abstract

Thermal noise arising from mechanical dissipation in oxide mirror coatings is an important limit to the sensitivity of future gravitational wave detectors, optical atomic clocks and other precision measurement systems. Here, we present measurements of the temperature dependence of the mechanical dissipation of an ion-beam sputtered silica film between 10 and 300 K. A dissipation peak was observed at 20 K and the low temperature dissipation was found to have significantly different characteristics than observed for bulk silica and silica films deposited by alternative techniques. These results are important for better understanding the underlying mechanisms of mechanical dissipation, and thus thermal noise, in the most commonly-used reflective coatings for precision measurements.

Keywords: gravitational waves, thermal noise, optical coatings, silica, mechanical dissipation, precision measurement

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1. Introduction

Long-baseline gravitational wave detectors use interferometric sensing to search for the displacements, induced by gravitational waves, of test-masses which are coated to form highly reflective mirrors and suspended as pendulums at the ends of perpendicular arms up to 4 km long. Brownian motion of the highly-reflective mirror coatings sets an important limit on the performance of these instruments, with coating thermal noise expected to limit the sensitivity of future generations of detectors in their most sensitive frequency band [1–4]. The magnitude of this thermal noise is directly related to the mechanical dissipation, or internal friction, of the amorphous oxide mirror coatings. Coating thermal noise can also significantly limit the performance of a range of ultra-precision experiments [5] including frequency-stabilized lasers [6], high resolution optical spectroscopy [7] and optical atomic clocks [8, 9].

The mirror coatings used in the first generation of gravitational wave detectors were formed from alternating layers of silica (SiO$_2$) and tantala (Ta$_2$O$_5$) deposited by ion-beam sputtering. Research has shown that the mechanical dissipation of these multi-layer coatings at room temperature is dominated by the dissipation of the tantala layers [10–12] and that the total dissipation of a multi-layer coating can be reduced by up to $\sim$40% by doping the tantala with titania (TiO$_2$) [13, 14]. However, further reductions in coating thermal noise are required to enable proposed future upgrades to the second generation detectors [15–17] and planned third generation detectors, such as the Einstein Telescope [18], to reach their planned sensitivities.

In general, lowering the operating temperature of the mirrors would be expected to reduce the magnitude of the thermal noise and some plans for upgrades to existing detectors involve the use of cryogenic cooling, either to 120 or 20 K [15, 16], while new detectors such as KAGRA [19] and the proposed Einstein Telescope are planned to operate in the range of 10–20 K. However, mechanical dissipation is often temperature dependent and previous studies have shown that the dissipation of ion-beam sputtered (IBS) tantala films increases at low temperatures, and have identified various peaks in the dissipation [20, 21]. Previous studies of the mechanical dissipation of multi-layer mirror coatings at cryogenic temperatures have been somewhat ambiguous, with measurements of silica/tantala multi-layer coatings by Yamamoto [22] at 20 K showing no evidence of the low-temperature dissipation peak which would be predicted from our studies of single layers of tantala [21]. However, more recent measurements of the Advanced LIGO multi-layer mirror coating [23], in which the tantala layers are doped with titania, did show a low temperature loss peak at a somewhat higher temperature ($\sim$26 K) than would be expected from our measurements of single layers of tantala. Clearly it is essential to characterize the dissipation of IBS silica coatings at cryogenic temperatures to develop a full understanding of the temperature-dependent dissipation in multi-layer coatings and thus enable accurate estimation of the level of coating thermal noise in future gravitational wave detectors. These studies are also important to allow calculations of the effect on the overall coating thermal noise of reducing the low-temperature loss of tantala (e.g. through optimized heat-treatment [21]) or of replacing tantala with possible alternative materials [24].

While fused silica in bulk form has been extensively studied and is known to exhibit a cryogenic dissipation peak centered on 30–40 K [25–28], there is relatively little literature on dissipation in thin silica films. One previous cryogenic study of silica films deposited by electron beam evaporation showed that they did not display the characteristic dissipation peak observed at approximately 30–40 K in bulk silica [29]. In contrast, films produced by the thermal oxidation of silicon for the same study did show a dissipation peak with identical characteristics to the peak observed in bulk silica. This result suggests that the method used to deposit a silica film, and thus the structure of the film, are strongly related to its low
temperature dissipation. The mechanical dissipation of IBS silica films has been studied at room temperature, where post-deposition heat-treatment has been observed to significantly lower the dissipation [30], possibly related to the reduction of the internal stress in the films.

Measurements of the temperature dependence of the mechanical dissipation in a material can provide insights into the microscopic mechanisms responsible for the dissipation of energy as described, for example, by Nowick and Berry [31]. Understanding the dissipation mechanisms in silica films is of particular interest as, at room temperature, the dissipation in IBS silica can be more than a factor of ten lower than that of tantala [10, 30].

2. Sample preparation

The mechanical dissipation of a thin coating can be calculated from the change in the mechanical dissipation of a resonator to which the coating is applied. In this study, the resonators were thin silicon cantilever structures, fabricated by a chemical etch from a (100) silicon wafer, of an identical design to previously measured samples [21] with the cantilever length along the [110] direction. The cantilevers had a 0.5 mm thick ‘clamping block’ at one end, which is designed to minimize the loss of energy into the support structure in which the cantilever is mounted. The flexing part of the cantilever is 50 μm thick. The low mechanical dissipation and high thermal conductivity of silicon at cryogenic temperatures makes these substrates particularly suitable for low-temperature studies of coating dissipation [32]. As the coating process involved thermal treatment steps which could potentially alter the mechanical dissipation of the silicon substrate, two nominally identical cantilevers were studied, one of which was coated while the other remained un-coated as a reference sample and was put through identical thermal treatments to the coated sample.

A 1 μm thick silica film was deposited by CSIRO using ion-beam sputtering at a temperature of 100 °C, with argon as the sputtering ion. To ensure good adhesion of the coating, a thin (~20 nm) thermal oxide layer was grown on the cantilever prior to coating using a dry oxidation process in which the cantilever was placed in an oven at 1000 °C for 30 min. Post-deposition heat-treatment at temperatures between 400 and 600 °C is usually carried out to relieve internal stresses and reduce the optical absorption of silica/tantala multi-layer coatings [33]. While the room temperature mechanical dissipation of silica films has been observed to be reduced by heat-treatment up to 900 °C [30], tantala films have been observed to crystallize at temperature above 600 °C [33]. Thus, the silica film studied here was heat-treated at 600 °C, to simulate the highest heat-treatment which could be carried out on a multi-layer silica/tantala coating without crystallization of the tantala layers. The cantilevers were heated to 600 °C at 2 °C min⁻¹ in an air environment and held at this temperature for 24 h before cooling overnight to 25 °C. This heat-treatment regime is identical to that used in our previous studies of single layers of tantala heat-treated at 600 °C.

3. Results

The temperature dependence of the mechanical dissipation of several resonant modes of both the coated and un-coated cantilevers was measured between 10 and 290 K in a cryostat using a ‘ring-down’ technique, in which a mode of the sample of angular frequency ω₀ was excited electrostatically and the dissipation ϕ(ω₀) found from the free exponential decay of the resonant motion, A(t) = A₀ e⁻ϕ(ω₀)ω₀²t/2. Full details of this experimental technique are discussed elsewhere [21, 34]. Several measurement cycles in which the sample temperature
was increased incrementally from approximately 10 to 290 K were carried out, with the sample being removed and re-clamped between cycles. Repeated ring-down measurements at each temperature generally showed a variation in dissipation of less than 5% for each mode. The variation in the dissipation between the various temperature cycles was typically less than 15%.

Figure 1 shows, as an example, the temperature dependence of the dissipation of a bending mode of the coated and un-coated samples at approximately 10.07 kHz. Also shown is the calculated level of thermoelastic dissipation, which arises from heat-flow through the silicon cantilever as it bends [35]. The increase in loss due to the silica coating can be observed throughout the temperature range, although close to room temperature the difference in loss between the two samples is small as the dissipation in both samples is dominated by thermoelastic dissipation in the silicon substrate. A peak in the loss of the un-coated sample was observed at 28–30 K in all of the modes and is consistent with the presence of thermal oxide on the cantilever surfaces [29]. The dissipation of the coated cantilever exhibits a peak at the lower temperature of ∼20 K, and an additional small peak at 160 K.

The mechanical dissipation of the silica coating can be calculated from the difference in the measured dissipation of the coated and un-coated cantilevers [34]:

\[
\phi(\omega_0)_{\text{coating}} = \frac{Y_s t_s}{3Y_c t_c}(\phi(\omega_0)_{\text{coated}} - \phi(\omega_0)_{\text{substrate}}),
\]

where \(\omega_0\) is the angular frequency of the mode, \(\phi(\omega_0)_{\text{coated}}\) is the loss factor of the coated cantilever, \(\phi(\omega_0)_{\text{substrate}}\) is the loss factor of the un-coated reference cantilever, \(t_s\) and \(Y_s\) are the thickness and Young’s modulus of the substrate respectively and \(t_c\) and \(Y_c\) are respectively the thickness and Young’s modulus of the coating. Young’s modulus of silicon and silica were taken to be 166 GPa [36] and 72 GPa [37], respectively. Young’s modulus of both materials varies by less than a few per cent over the temperature range studied [27, 36] and have therefore been assumed to be constant.

Equation (1) was used to calculate the mechanical dissipation of the silica coating for five modes of the cantilever between 1.1 and 16 kHz. As shown in figure 2, the dissipation of the silica coating has a broadly similar trend for each of the modes, with dissipation peaks observed...
Temperature dependence of the mechanical dissipation of the silica coating, calculated from the measured mechanical dissipation of each mode of the coated and un-coated cantilever samples.

Figure 2. Temperature dependence of the mechanical dissipation of the silica coating, calculated from the measured mechanical dissipation of each mode of the coated and un-coated cantilever samples.

at 20 and 160 K, as expected from the measured loss of the coated sample (see figure 1), and a third peak at approximately 250 K. This peak was not readily apparent in the measurements of the coated cantilever due to the masking effect of the rapidly rising loss of the cantilever substrate in this temperature range.

For both the loss peak at ∼20 K and at ∼250 K, the temperature of the peak was observed to increase with mode frequency. This behavior is characteristic of a thermally activated dissipation process which can be characterized by a rate constant, $\tau_0$, and an activation energy, $E_a$, related by the Arrhenius equation [31]:

$$\tau = \tau_0 e^{E_a/k_B T},$$

where $\tau$ is the relaxation time associated with the dissipative system returning to equilibrium after a perturbation. Analysis of these dissipation processes shows that the temperature of the dissipation peak, $T_{\text{peak}}$, at angular frequency $\omega_0$ is related to the activation energy and rate constant as follows [31]:

$$\omega_0 \tau_0 e^{E_a/k_B T_{\text{peak}}} = 1.$$  

Thus, the activation energy and rate constant for the dissipation process can be calculated from a linear fit to a plot of $\ln \omega$ against $1/T_{\text{peak}}$. Figure 3 shows this analysis for the peak near 20 K. The linear fit has a coefficient of determination of 0.96. The activation energy and rate constant were calculated from the fit to be $(32.1 \pm 2.9) \text{ meV}$ and $(1.8 \pm 0.1) \times 10^{-13} \text{ s}$, respectively. Interestingly, this activation energy is somewhat lower than observed for bulk fused silica, as will be discussed further below. The activation energy is very similar to that found for un-doped tantala films under the same heat-treatment conditions as the silica film studied here, although it should be noted that the loss peak observed in tantala films is significantly narrower than the silica peak [20].

An Arrhenius analysis was also carried out for the dissipation peak at 250 K, as shown in figure 4. It should be noted that both sides of the peak were not fully resolved for the mode at 12 kHz resulting in a large error in the position of this peak. This mode was therefore initially excluded from the analysis. The activation energy and rate constant were found to be $(0.43 \pm 0.11) \text{ eV}$ and $(5.3 \pm 0.8) \times 10^{-14} \text{ s}$, respectively. The coefficient of determination for
Figure 3. Arrhenius plot for the loss peak observed around 20 K.

Figure 4. Arrhenius plot for the loss peak observed around 250 K.

this Arrhenius fit was 0.84. Inclusion of the 12 kHz mode in the linear fit does not significantly change these values.

No correlation was observed between mode frequency and peak temperature for the dissipation peak at 160 K. While we have previously observed excess loss in individual modes associated with resonant coupling of energy between the cantilever and the clamping structure [38], the fact that the 160 K peak is present in all of the modes studied, over a frequency range of 15 kHz, tends to rule out this explanation. Clearly further investigation of this peak is of interest.

4. Analysis and discussion

The dissipation peak at 20 K is perhaps the most interesting feature of the data, both in terms of relevance to future gravitational wave detectors and when compared to previous measurements
Figure 5. Comparison of the mechanical dissipation of ion-beam sputtered silica with bulk silica [41], e-beam evaporated silica and thermal oxide grown on silicon [29].

... of the dissipation of silica. The dissipation in bulk samples of silica has been extensively studied as a function of temperature and a dissipation peak centered on approximately 40–60 K has been well characterized by many authors [25, 39–41]. White and Pohl have studied the dissipation of various thicknesses of silica film deposited by electron-beam evaporation and of a 500 nm thick film produced by the thermal oxidation of silicon substrates [29, 42]. These measurements were carried out at 5.5 kHz between 10^{-2} and 100 K. The dissipation of the thermal oxide was found to closely resemble that observed in bulk silica samples, with a dissipation peak at 35 K, while the e-beam films were observed to have very different behavior. Figure 5 shows a comparison of the low-temperature dissipation measured here in IBS silica at 7 kHz with the loss of bulk silica at 56 kHz [41] and with the loss of the thermal oxide film and various e-beam silica films measured by White and Pohl [29].

This comparison shows several interesting features. The very thin e-beam evaporated films studied by White and Pohl at temperatures up to 20 K have a similar magnitude of loss to the IBS film. However, the 109 nm thick e-beam film was observed to have higher loss between 10–20 K and to display very different temperature-dependent behavior from bulk, thermal oxide and IBS silica, with the loss increasing rapidly perhaps toward a peak at a temperature above 100 K. White and Pohl suggest that macroscopic disorder occurring in thicker e-beam films, perhaps related to porosity, may be responsible for the low-temperature dissipation of this film. IBS coatings tend to be more dense and have better stoichiometry than e-beam deposited films [43] due to the higher sputtering energy, and it is possible that the different temperature-dependent dissipation is related to structural differences associated with the deposition method. It is interesting to note that the 1 μm thick IBS film has a similarly low level of loss as the thinnest (sub-10 nm) e-beam films. This may suggest that the macroscopic disorder which arises as thicker e-beam films are grown is not be present in IBS films, which is consistent with dense, non-porous structure of IBS films.

The magnitude of the dissipation of the IBS film is lower than that of the bulk silica and the thermal oxide at temperatures below 85 K, and the dissipation peak in the IBS film occurs at a lower temperature of 18–20 K than the peak observed in thermal oxide and bulk silica at approximately 35 K. Comparison of the peak temperature is valid for the IBS and the
thermal oxide films, which were measured at very similar frequencies. Since the temperature of the dissipation peak depends on the frequency at which the measurement was carried out, Cahill and van Cleve’s measurements of the dissipation of bulk silica at 56 kHz are not directly comparable to the measurements of the IBS films. However, equation (2) can be used to extrapolate the position of the bulk dissipation peak at lower frequencies. At the lowest frequency studied here (1.1 kHz), this extrapolation predicts the peak in bulk silica to occur at 26 K: still significantly higher in temperature than the peak at 18.5 K observed in the IBS film at this frequency. Indeed, this type of extrapolation predicts that the dissipation peak in bulk silica will only occur at temperatures below 20 K at vibrational frequencies of less than 1 Hz [44]. The activation energy of (32.1 ± 2.9) meV found for the dissipation mechanism in the IBS film is somewhat lower than the activation energy of 44–52 meV found in different studies for the dissipation peak in bulk silica [25, 44].

In common with many amorphous materials [45], the dissipation peak in silica is wider than would be expected for a dissipation process with a unique activation energy, and the width of the peak is thought to be associated with the disordered structure of the silica giving rise to a distribution of activation energies. Several models of the microscopic mechanism responsible for the low-temperature dissipation peak in fused silica have been proposed, including motion of Si-O bonds between stable angles [25], elongation of Si-O bonds [39], rotation of SiO$_4$ tetrahedral structures [46, 47] or of groups of these structures [48]. All of these mechanisms essentially involve bonds reorienting over potential barriers, and the dissipation can be modeled as arising from transitions in an asymmetric double-well potential in some local configuration coordinate [49]. In this case, the activation energy calculated from an Arrhenius analysis represents the average barrier height in this distribution. It can be shown that the dissipation is related to the distribution of barrier heights $g(V)$ by the following expression [50]:

$$
\phi = \frac{\pi \gamma^2 f_0}{C_{ii}} k_B T g(V),
$$

(4)

where

$$
V = k_B T \ln \left( \frac{1}{\omega \tau_0} \right)
$$

(5)

and $C_{ii}$ is the appropriate elastic constant for the mode-shape under consideration, $\gamma$ is the elastic coupling constant which represents the coupling between the defect (e.g. a bond re-orienting within the double well potential) and the applied strain and $f_0$ is a constant representing the asymmetry between the depth of the two potential wells in each double-well system. The relaxation time $\tau$ associated with a barrier height $V$ is given by equation (2). The detailed assumptions made in this model are discussed by Topp and Cahill [50], and the values of $C_{ii}$ and $\gamma$ for fused silica ($3.3 \times 10^4$ and 0.9 eV respectively) were taken from this paper. Figure 6 shows the distribution of barrier heights associated with the 20 K peak in the IBS silica film, calculated with equation (4) using the measured dissipation at 7 kHz. This analysis uses only data measured at temperatures below 80 K; in this temperature range, it seems very likely that the dissipation in this temperature range is dominated by the tail of the 20 K peak. There is excellent agreement in both the magnitude and shape of the barrier height distribution calculated from four of the modes, with no apparent frequency dependence. However, the magnitude of the distribution for the 1.1 kHz mode was larger than for all of the other modes. This is a direct consequence of the higher coating dissipation measured for this mode, and may be related to frictional losses associated with the clamp at this frequency, or to interactions with another mode of the cantilever, believed to be a torsional mode, at the nearby frequency of 1178 Hz. The barrier height distribution for bulk fused silica was also calculated using the dissipation measurements by Cahill and van Cleve in figure 5.
While this extraction of the barrier heights is simply a re-scaling of the mechanical dissipation data, it can provide a useful illustration of the differences in the dissipation peaks observed in bulk silica and in the IBS silica film. The magnitude of the distribution \( g(V)f_0 \) is lower in the case of IBS silica at barrier energies below 100 meV, although at higher energy the two distributions begin to converge. This suggests that there are few low-energy barriers contributing to the dissipation in the IBS silica than in the bulk case. It may be possible that the structure or the internal stress of the IBS film alters the distribution of barrier heights from the bulk case, for example by changing the distribution of Si-O bond angles. From measurements of the radius of curvature of the coated silicon cantilever [51], the stress in the silica film was calculated to be \((223\pm13)\) MPa. Studies of the amorphous structure of thin-films using transmission electron microscopy have begun to identify correlations between local atomic structure and mechanical dissipation in tantala films at room temperature [52]. The application of these techniques to compare the structure of bulk silica and IBS silica films may be of significant importance in fully understanding the differences in the dissipation observed. The effect on the loss of the sputtering parameters used when depositing the film may also be an interesting area for future investigations.

Our measurement of a dissipation peak at approximately 20 K in IBS silica is of direct interest to future cryogenic gravitational wave detectors. The peak is at an almost identical temperature to the dissipation peak we have previously identified in tantala coatings under identical heat-treatment conditions [20]. The magnitude of the peak in silica varied between \(5 \times 10^{-4}\) and \(9 \times 10^{-4}\), while the peak in tantala was found to vary between \(8 \times 10^{-4}\) and \(1.2 \times 10^{-3}\). These results indicate that the loss of the silica layers in silica/tantala multi-layer coatings is likely to begin to contribute significantly to the total coating loss at temperatures below 50 K, and may contribute equally with the tantala layers at temperatures around 20 K. While at room temperature efforts to reduce the multi-layer coating loss have focused on reducing the loss of the tantala layers, at cryogenic temperature it will also be necessary to either reduce the loss of the silica layers or identify a suitable alternative material of lower mechanical dissipation.
Although the dissipation peaks observed at 160 and 250 K are not in the temperature ranges under consideration for the operation of future gravitational wave detectors, their origin is also of interest. Similar peaks do not appear to have been observed in bulk silica, although a broad plateau in the dissipation has been observed at approximately 200 K [53] and two dissipation peaks at much higher temperatures (approximately 1000 and 1440 K) are also known to occur [44]. It is possible that the dissipation of IBS silica films at room temperature is limited by the trailing edge of the peak at 250 K. The mechanical dissipation of bulk silica at room temperature can be reduced by using heat-treatment to reduce the internal stress [54, 55]. IBS coatings generally have relatively high levels of stress in comparison to coatings deposited using alternative techniques [56], and studies of the mechanical loss of IBS silica films at room temperature have shown that the mechanical dissipation can be significantly also reduced by heat-treatment [30]. Thus, the loss peak at 250 K, which appears to limit the loss of IBS silica at room temperature, may be related to stress in the coating, and further studies on the effect of heat-treatment on this dissipation peak would be of interest.

5. Conclusions

We have measured the mechanical dissipation of an ion-beam sputtered silica coating and observed three dissipation peaks at approximately 20, 160 and 250 K. The 20 K dissipation peak is of a similar magnitude to the dissipation peak observed in tantala films under the same heat-treatment conditions, suggesting that, in the silica/tantala coatings currently used for gravitational wave detectors, both materials will contribute significantly to the total mechanical loss of the coating at low temperatures. This dissipation peak is at a significantly lower temperature, and of a lower activation energy, than the peak observed in bulk silica and thermal oxide, suggesting that the loss mechanism in IBS silica may be different from the mechanism responsible for the loss in bulk silica and thermal oxide. The differences in the behavior of IBS silica, and bulk silica/thermal oxide may give key insights into understanding mechanisms responsible for mechanical dissipation, and hence thermal noise, in thin films. Better understanding of the processes responsible for mechanical dissipation in IBS films is likely to be critical in identifying methods by which the mechanical dissipation, and thus Brownian thermal noise, can be further reduced in a wide range of ultra-precision measurements.

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