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# Comparison of the temperature dependence of the mechanical dissipation in thin films of $Ta_2O_5$ and $Ta_2O_5$ doped with $TiO_2$

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**Abstract.** Here we report the first results comparing the temperature dependence of the mechanical dissipation in thin films of $Ta_2O_5$ and $Ta_2O_5$ doped with $TiO_2$, of a type suitable for use in the multilayer optical coatings for advanced gravitational wave detectors. The results indicate that doping $Ta_2O_5$ with $TiO_2$ can significantly alter the distribution of activation energies associated with the low temperature dissipation peak.

## 1. Introduction

Long-baseline gravitational wave detectors are used in searches for gravitational radiation from astrophysical sources. Interferometric sensing is employed to monitor the displacements of test-masses, which are coated to form highly reflective mirrors and suspended as pendulums at the ends of perpendicular arms. The thermal noise

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associated with the test mass mirrors and their suspensions forms a significant limit to the detectors sensitivity [1]. In particular, the mechanical dissipation of the ion-beam-sputtered test mass mirror coatings has been identified as an important noise source which is likely to limit the sensitivity of the next generation of detectors in their most sensitive frequency band [2, 3, 4, 5].

The mirror coatings used in current gravitational wave detectors are formed from alternating layers of ion-beam sputtered silica ($SiO_2$) and tantalum pentoxide ($Ta_2O_5$). Research has shown that the mechanical dissipation of these multilayer coatings is dominated by the dissipation of the $Ta_2O_5$ layers [6, 7, 8] and that the dissipation can be reduced by up to $\sim 40$ % by doping the $Ta_2O_5$ with titania ($TiO_2$) [9]. However, neither the process responsible for mechanical dissipation in $Ta_2O_5$ or the mechanism by which $TiO_2$ doping reduces the dissipation is clearly understood.

In a recent paper we reported data showing a low temperature dissipation peak in a $Ta_2O_5$ coating doped with $TiO_2$ and annealed at 600 °C [10]. There is evidence that this peak arises from a thermally activated dissipation mechanism, possibly related to the reorientation of Ta-O bonds within a double well potential, similar to the mechanism believed to occur in fused silica [11, 12, 13]. It is therefore of significant interest to compare the temperature dependence of the dissipation of doped and undoped tantala coatings in order to investigate the effects of doping on the behaviour of this dissipation peak.

## 2. Sample preparation and experimental technique

The mechanical loss of a coating layer applied to a substrate can be calculated from the difference in the loss of the substrate before and after deposition of the coating. In this study rectangular silicon cantilevers, described in detail elsewhere [10, 14], were used as coating substrates. The cantilevers were fabricated by a chemical etch and comprised a 0.5 mm thick 'clamping block', used to mount the cantilever between stainless steel blocks, and a flexing part 34 mm long and 47.5 ± 0.5 μm thick. Silicon is a suitable substrate for these studies as it is known to have a low mechanical loss at low temperature [15, 16] thus minimising the contribution of the substrate to the total measured loss.

For the bending modes of these cantilever samples the loss of the coating layer, $\phi(\omega_0)_{\text{coating}}$, can be shown to be given by [17]:

$$\phi(\omega_0)_{\text{coating}} = \frac{Y_{\text{s}} t_s}{3 Y_{\text{c}} t_c} (\phi(\omega_0)_{\text{coated}} - \phi(\omega_0)_{\text{substrate}}), \tag{1}$$

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 cantilever prior to coating, $t_s$ and $Y_{\text{s}}$ are the thickness and Young's modulus of the substrate respectively and $t_c$ and $Y_{\text{c}}$ are respectively the thickness and Young's modulus of the coating.

A single layer of $Ta_2O_5$ of 0.5 μm in thickness was applied to a silicon cantilever substrate by ion beam sputtering. It should be noted that the coating process involved

two heat treatment steps which could potentially change the loss of the silicon cantilever itself. Firstly, to ensure proper adhesion of the coating, a thermal oxide layer of thickness approximately 20 to 30 nm was grown on the cantilever surface. Secondly, after deposition of the coating, the cantilever was put through a heat treatment procedure at 600°C for 24 hours under atmospheric conditions. Post-deposition heat treatment is standard practice to reduce absorption losses and to relieve the internal stress in ion-beam sputtered multilayer coatings [18]. To account for any possible effect on the loss of the silicon substrate, an identical cantilever was oxidised and put through the same heat treatment process, without being coated with $Ta_2O_5$. The coating loss could then be calculated from the difference in the loss factors of the coated and uncoated cantilevers.

## 3. Results

The mechanical loss factors of the first five bending modes of the coated cantilever were measured between approximately 11 K and 300 K, using a 'ring-down' technique as described (for example) in [14]. The mode frequencies studied ranged from 55 Hz to 3.2 kHz. The temperature of the cantilever was measured using a silicon diode sensor mounted within the clamp immediately below the fixed end of the cantilever. Several measurement cycles, in each of which the sample temperature was increased incrementally from 11 to 300 K, were carried out. Several ringdown measurements of the modes under study were made at each temperature step in these cycles. The variation in the loss factor was typically found to be significantly less than 10 %.

The mechanical loss factor of the $Ta_2O_5$ coating was calculated using Equation 1. The loss of the uncoated substrate, required for this calculation, had been measured for an identical silicon cantilever which had undergone the same thermal oxidation and heat treatment as the cantilever coated with $Ta_2O_5$ [10]. The Young's modulus of the $Ta_2O_5$ coating was taken to be $140 \pm 15$ GPa [19]. A peak in the dissipation of the coating was observed for each mode. Figures 1 to 5 show a comparison of the dissipation peak found in the undoped coating studied here and the $TiO_2$-doped coating [10] for the first five bending modes of the samples. In Figure 6 the dissipation of the two coatings calculated for the third mode at $\sim$ 1000 Hz is compared throughout the entire temperature range studied. In these plots, the errors bars reflect the total uncertainty in the calculated coating loss, arising from both the experimental error in the measured loss factors and the uncertainties in the Young's modulus and thickness of both the coatings and the cantilever substrates. It should be noted that the uncertainty in the Young's modulus of the coating is the most significant contribution to the total error in the coating loss, typically accounting for $\sim$50 % of this error.

In Figures 1 to 5 it can be seen that the peak in the undoped coating is narrower than the peak observed in the coating doped with $TiO_2$ and it is clear from a fit to a Debye peak [20] that the loss peak in the undoped coating occurs at slightly lower temperatures. No significant change in the temperature of the loss peak was observed when the sample was re-clamped. The peak dissipation is higher for the undoped coating

for four of the five modes studied. For the third mode at $\sim$ 1000 Hz, the peak dissipation was approximately $1.1\times10^{-3}$ for both coatings, as shown in Figure 3. However, it should be noted that the dissipation peak in the doped coating exhibited some scatter at this frequency and was significantly higher than was observed for all of the other modes [10].

At the wings of the peak (i.e. in the temperature ranges $\sim$ 11-18 K and $\sim$ 22-40 K), the loss of the undoped coating was slightly lower than that of the doped coating. Further investigation of the losses at these temperatures may be of interest. However, throughout the remainder of the temperature range studied the undoped coating tends to have a higher loss, as shown in Figure 6.

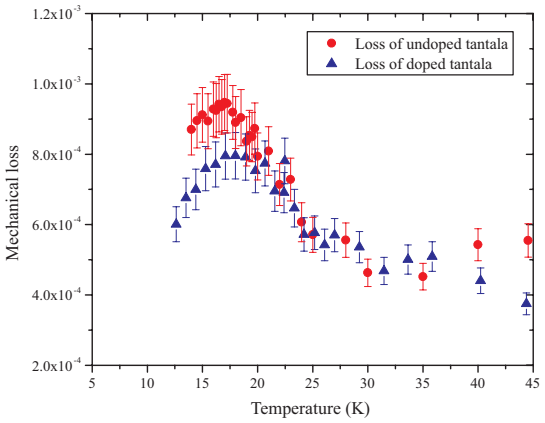


**Figure 1.** *Comparison of the loss peak of an undoped $Ta_2O_5$ and a $Ta_2O_5$ coating doped with $TiO_2$ at $\sim$ 55 Hz.*

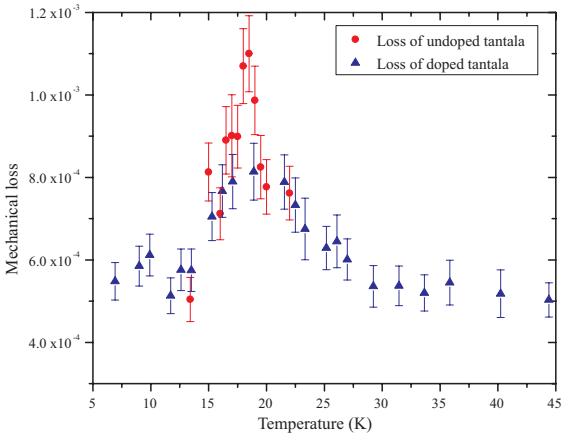


**Figure 2.** *Comparison of the loss peak of an undoped $Ta_2O_5$ and a $Ta_2O_5$ coating doped with $TiO_2$ at $\sim$ 350 Hz.*

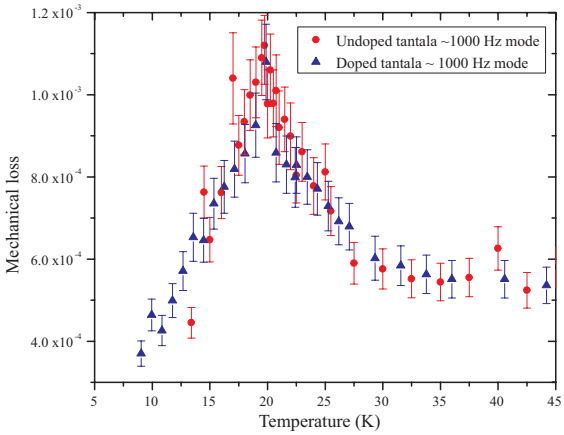


**Figure 3.** *Comparison of the loss peak of an undoped $Ta_2O_5$ and a $Ta_2O_5$ coating doped with $TiO_2$ at $\sim$ 1000 Hz.*

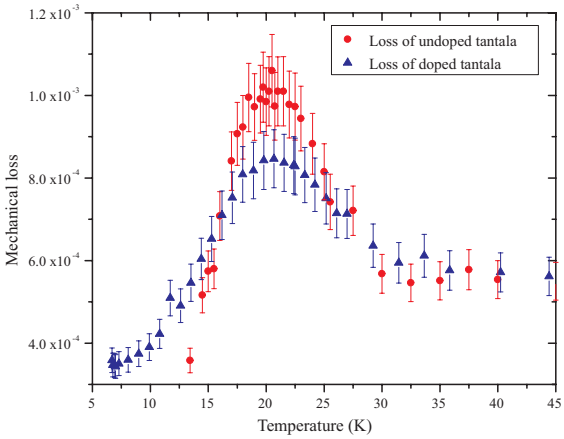


**Figure 4.** *Comparison of the loss peak of an undoped $Ta_2O_5$ and a $Ta_2O_5$ coating doped with $TiO_2$ at $\sim$ 1900 Hz.*

## 4. Analysis

The temperature $T_{\rm peak}$ at which the peak dissipation occurs is frequency dependent. This is typically indicative of a thermally activated relaxation process. Analyses of these processes has shown that they follow the relation [21]:

$$\omega_0 \tau_0 e^{E_a/k_B T_{\rm peak}} = 1, \tag{2}$$

where $\omega_0$ is the angular frequency of the resonant mode under study, $\tau_0$ is the relaxation constant and $E_{\rm a}$ is the activation energy of the relaxation process. Plotting the natural logarithm of the angular frequency against $1/T_{\rm peak}$ gives a straight line with slope $E_{\rm a}/k_{\rm B}$. Such a plot - known as an Arrhenius plot - for the peak observed in the undoped coating is shown in Figure 7. An activation energy of $E_{\rm a} = 28.6 \pm 1.2$ meV and a relaxation constant of $\tau_0 = 5.9 \pm 0.2 \times 10^{-12}$ s were calculated from the slope of this line.

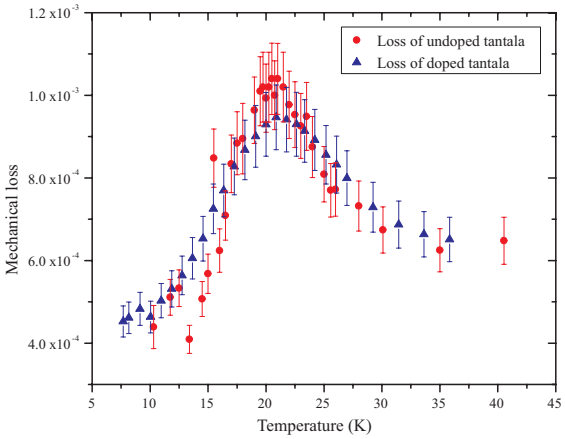


**Figure 5.** *Comparison of the loss peak of an undoped $Ta_2O_5$ and a $Ta_2O_5$ coating doped with $TiO_2$ at $\sim$ 3200 Hz.*

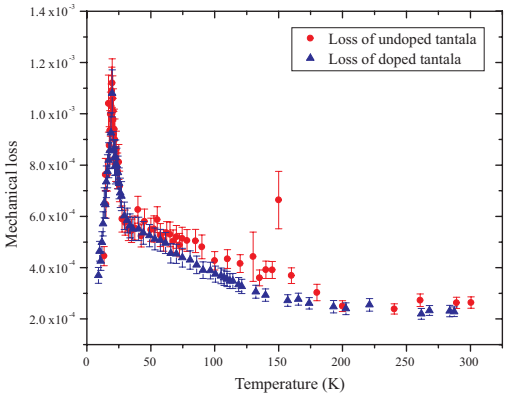


**Figure 6.** *Comparison of the loss of an undoped $Ta_2O_5$ and a $Ta_2O_5$ coating doped with $TiO_2$ at $\sim$ 1000 Hz.*

The dissipation peak is significantly broader than would be expected if the dissipation arose from a process with a unique activation energy. This is characteristic of amorphous materials [22, 23] and implies that there is a broad distribution of activation energies, with an average value of $E_a = 28.6 \pm 1.2$ meV. Also shown in Figure 7 is the data for the doped coating, for which the activation energy and relaxation constant were $39.5 \pm 2.7$ meV and $1.7 \pm 0.1 \times 10^{-14}$ s respectively. The results suggest that doping $Ta_2O_5$ with $TiO_2$ increases the average value of the activation energy of the process responsible for the dissipation by approximately 33 % and decreases the relaxation constant by approximately two orders of magnitude.

Mechanical dissipation peaks occurring at temperatures above 10 K in amorphous solids [24], such as fused silica, are thought to arise from thermally activated transitions of atoms or molecules between stable orientations [11]. This can be represented by a double well potential, with two minimum energy states separated by a potential energy

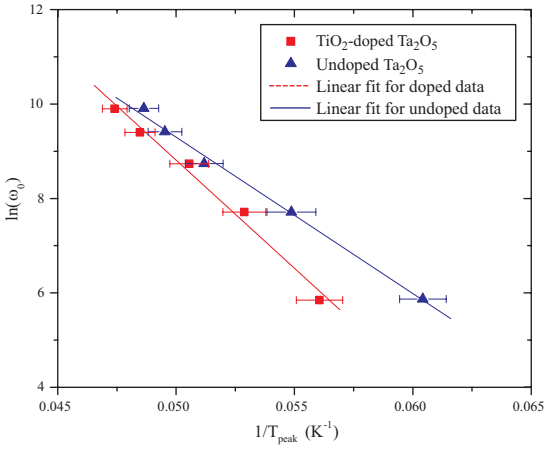


**Figure 7.** *Comparison of the Arrhenius plots of $ln(\omega_0)$ against $1/T_{peak}$ for the dissipation peaks in $Ta_2O_5$ doped with $TiO_2$ and undoped $Ta_2O_5$.*

barrier of height $V$ [23]. The amorphous nature of these materials results in a wide distribution of potential barrier heights, explaining the broad nature of the dissipation peaks observed in these materials. Initial models were based on the assumption that the double-well potentials were symmetric, with the potential barrier separating states of equal potential energy [13, 23, 25, 26]. Gilroy and Phillips [27] proposed a refined version of this model in which the potential well depths are asymmetric, as a result of the same structural variations which give rise to the distribution of potential barrier heights. In fused silica, this model was found to explain the observed linear scaling of the peak loss with the measurement frequency [27]. This model has also been shown to account for the anomalous low-temperature specific heats and thermal conductivities in disordered materials [28].

Figure 8 shows a model of an asymmetric double-well potential (ADWP), with a barrier height $V$ and an asymmetry $\Delta$ in the energies of the wells. In an amorphous solid there will be a distribution of potential barrier heights, $g(V)$, and also a distribution

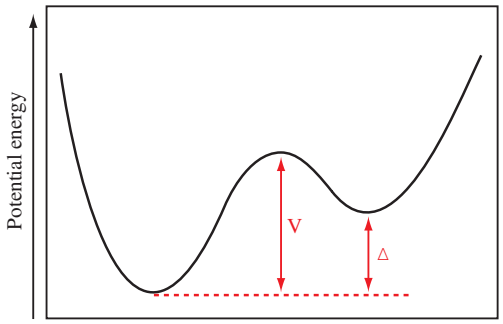


**Figure 8.** *A schematic diagram of an asymmetric double well potential, with a potential barrier $V$ and an asymmetry $\Delta$.*

$f(\Delta)$ of the asymmetries in the energies of adjacent potential wells. It can be shown that the dissipation predicted by this model is given by the following double integral over $\Delta$ and $V$ [27]:

$$\phi = \frac{\gamma^2}{k_B T C_{\rm ii}} \int_0^\infty \int_0^\infty \frac{\omega\tau}{1+(\omega\tau)^2} \mathrm{sech}^2\left(\frac{\Delta}{2k_{\rm B}T}\right) f(\Delta) g(V) d\Delta dV, \tag{3}$$

where $\omega$ is the angular frequency of measurement, $C_{\rm ii}$ is the appropriate elastic constant, $\gamma$ is the elastic coupling constant which represents the coupling between the defect (e.g. the atom re-orienting within the ADWP) and the applied strain. The relaxation time $\tau$ associated with a barrier height $V$ is given by the Arrhenius equation [21].

Both the barrier height distribution $g(V)$ and the barrier asymmetry distribution $f(\Delta)$ are dependent on the microscopic structure of the amorphous film, which will not change in the temperature range studied, which is far below the glass transition temperature. Therefore it is assumed that $g(V)$ is independent of temperature and that $f(\Delta) = f_0 = \text{const.}$ [27]. Thus Equation 3 can be simplified to give the following expression relating the mechanical loss to the function $g(V)$ [29]:

$$\phi = \frac{\pi\gamma^2 f_0}{C_{\rm ii}} k_{\rm B} T g(V), \tag{4}$$

where

$$V = k_{\rm B} T \ln\left(\frac{1}{\omega\tau_0}\right). \tag{5}$$

Thus the distribution of barrier heights, $g(V)$, can be calculated from the measured temperature dependence of the mechanical loss. This calculation was carried out for the doped and undoped tantala loss data using the values of the relaxation constant, $\tau_0$, obtained from the Arrhenius fits: $\tau_0 = 5.9 \times 10^{-12}$ s for undoped tantala and $\tau_0 = 1.7 \times 10^{-14}$ s for doped tantala. Presented here are the results for the fourth bending mode at approximately 1900 Hz. The data for the other modes show similar results. The data used for the calculation are shown in Figure 9.

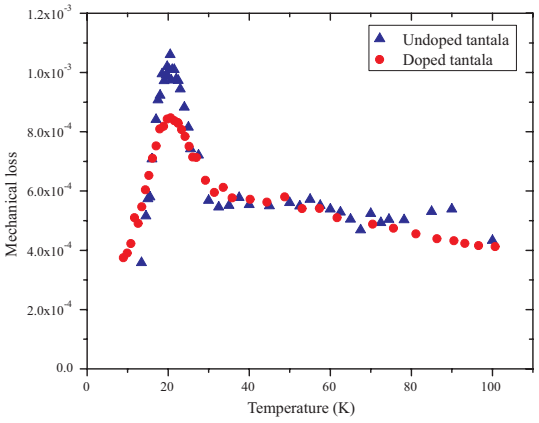


**Figure 9.** *Plot of the loss data for the fourth mode at ∼ 1900 Hz, used to calculate the barrier height distribution functions.*

The value of the coupling coefficient $\gamma$ for $Ta_2O_5$ is not known. However for most amorphous solids $\gamma$ lies in the range $0.6\text{eV} < \gamma < 1.1$ eV [29]. For the present analysis the value of $\gamma$ given for fused silica, $\gamma = 0.9$ eV, was used. Data for the elastic constants $C_{ii}$ of various amorphous materials are given in Topp and Cahill [29], all falling in the range $1.4 \times 10^4 < C_{ii} < 7.8 \times 10^4$. $C_{ii}$ for both doped and undoped $Ta_2O_5$ was approximated by scaling the value for silica, $C_{ii} = 3.3 \times 10^4$ [30], by the ratio of the Young's modulii of $Ta_2O_5$ and silica, giving $C_{ii} = 6.42 \times 10^4$. It should be noted that, while we have used estimated values of $C_{ii}$ and $\gamma$, these constants are likely to be similar for both doped and undoped tantala, thus the precise values are of little importance for comparing the two materials. Furthermore, the constants scale the absolute magnitude of the barrier height distribution but not its shape, which is of primary interest.

The calculated functions $g(V)f_0$ for both tantala coatings are shown in Figure 10. At high barrier energy, the barrier height distribution $g(V)f_0$ can be seen to decrease for both coatings. Both of the tantala coatings have a peak in the barrier height distribution. The presence of $TiO_2$ doping appears to shift the peak in the distribution to a higher potential barrier energy, and to reduce the height of the peak by approximately 16 %. In addition, the $TiO_2$ doping appears to increase the width of the peak in the distribution function. If, as we have postulated, the dissipation mechanism is related to structural relaxations involving the Ta-O bonds, one possible model is that the $TiO_2$ molecules can occupy a vacancy in the $Ta_2O_5$ structure, thus increasing the potential barrier to be overcome for reorientation of a bond.

The distribution of energy barriers is of interest in interpreting the effect of doping on the mechanical loss factors of the coatings. The doped coating has a larger number of high energy barriers suggesting that the loss factors of the doped coating would be expected to be lower, as is observed.

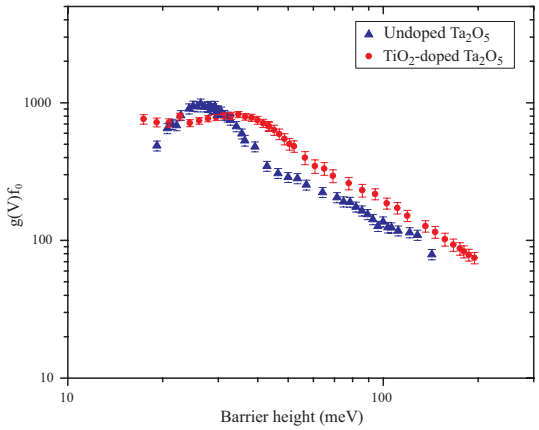


**Figure 10.** *Comparison of the calculated potential barrier height distribution function $g(V)f_0$ as a function of barrier height for undoped tantala and tantala doped with titania, using the data measured at $\sim$ 1900 Hz.*

## 5. Conclusions

The temperature dependence of the mechanical dissipation of an ion-beam sputtered $Ta_2O_5$ coating has been measured between 11 and 290 K, showing a peak in the dissipation around 20 K with an associated activation energy of 28.6 $\pm$ 1.2 meV. Comparison to our previous results for a $Ta_2O_5$ coating doped with 14.5% $TiO_2$ shows that the presence of $TiO_2$ reduces the height and increases the width of the dissipation peak, and significantly increases the average activation energy of the associated dissipation mechanism.

A barrier height distribution function analysis revealed that the effect of $TiO_2$ doping was to shift the distribution of potential barriers to a higher energy, to broaden the peak in the distribution and to reduce the height of the peak. This indicates that further reductions in mechanical loss, important for the reduction of coating thermal noise in gravitational wave detectors, may be possible if the distribution of barrier heights could be shifted to a higher energy. Thus the effect of the concentration and type of doping on the mechanical dissipation at low temperatures may be of interest for further investigation. Furthermore, it is known that in fused silica heat treatment can significantly reduce the mechanical loss [31, 32, 33]. This is believed to be a result of a reduction of stress in the silica, possibly altering the distribution of the barrier heights. Thus investigating the effects of heat treatment on the mechanical loss of tantala, with a view to reducing the coating mechanical loss, is of particular interest. It should be noted that at temperatures greater than $\sim 600$°C the optical properties of $Ta_2O_5$ may degrade, thus these properties must also be considered when evaluating the effects of heat treatment.

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