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Low-temperature mechanical dissipation of thermally evaporated indium film for use in interferometric gravitational wave detectors

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Abstract

Indium bonding is under consideration for use in the construction of cryogenic mirror suspensions in future gravitational wave detectors. This paper presents measurements of the mechanical loss of a thermally evaporated indium film over a broad range of frequencies and temperatures. It provides an estimate of the resulting thermal noise at 20 K for a typical test mass geometry for a cryogenic interferometric gravitational wave detector from an indium layer between suspension elements.

Keywords: indium, thermal evaporation, thin film coatings, thin film deposition, low temperature physics, gravitational wave detectors, gravitational waves

(Some figures may appear in colour only in the online journal)

1. Introduction

Interferometric gravitational wave detectors search for displacements, resulting from gravitational waves, of highly reflective mirrors suspended as pendulums at the end of kilometer-scale perpendicular arms [1]. Thermal noise associated with the mirrors and their suspensions will form a significant limit to these detectors [2]. To reduce thermal noise, the use of cryogenic cooling has been proposed for updates to existing detectors and for new detectors such as KAGRA [3], designed to operate at 20 K, and the proposed Einstein telescope (ET) low frequency detector [4–7] at 10 K. The fused silica mirrors used currently in room temperature detectors are not suitable for cryogenic operation due to a large increase in the mechanical dissipation in silica around 40 K [8–10]. Crystalline materials such as sapphire and silicon are known to have low mechanical dissipation at low temperature [11, 12] and these materials are part of the baseline design for both KAGRA and ET respectively [3, 13].

Various approaches to suspending such crystalline test masses are under consideration including the use of hydroxide-catalysis bonding [14], indium bonding or possibly a combination of both techniques to fabricate as much of a rigid quasi-monolithic suspension structure as possible [15–17]. Indium's low melting point of 156.6°C would allow relatively easy de-bonding of indium jointed components should something break or need to be replaced during the assembly or the operation of a cryogenic detector.

Indium bonding is a commonly used technique at low temperature, particulary for packaging microelectromechanical systems, due to its high heat transmission and good electrical contact [18] but not where thermal noise is important. Small scale indium bonds on 200 μm wide discs have demonstrated that, at 77 K, tensile strengths of around 20 MPa are achievable [18], a factor of three less than hydroxide-catalysis bonds between sapphire at the same temperature [14]. However, before this technique is employed, it is essential that investigations are made into the levels of mechanical loss, thermal conductivity and the strength of indium at the temperature at which a future cryogenic interferometric detector will operate.

Historically, several different experiments to determine the mechanical loss of wire slung suspensions, hydroxide catalysis bonded suspensions and joints formed with indium have also been investigated [19–23]. Investigations of a cylindrical quartz mass supported via an indium joint onto a flat on the top surface of the mass showed that the loss values obtained were similar to the intrinsic levels of loss of the mass when it was supported in a wire sling [19]. The Advanced LIGO interferometric gravitational wave detectors currently under construction use a mixture of wire on the upper stages, hydroxide catalysis bonding and the welding of silica components to create the quasi-monolithic suspensions which support each of the test masses [24, 25].

Liu *et al* [26] have previously measured the mechanical loss of one resonant mode of an e-beam evaporated indium film on an aluminium double-paddle oscillator as part of a study of metal films down to ~4 K. In this paper, mechanical loss measurements of multiple frequencies for a thin film of thermally evaporated indium are presented, enabling a preliminary estimate to be made of the thermal noise at 20 K in an interferometric gravitational wave detector from an indium layer used to joint suspension elements to the mirror.

2. Cantilever preparation

The mechanical loss of a thin film can be calculated from the change in the mechanical dissipation of a silicon cantilever caused by the addition of a film to its surface. Silicon is a

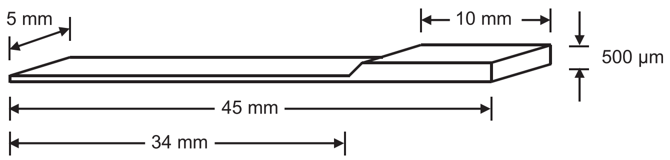

Figure 1. A schematic diagram of a silicon cantilever (not drawn to scale).

particularly useful substrate for use in studies of thin film dissipation at low temperatures due to its low mechanical dissipation and high thermal conductivity [27]. The thin silicon cantilevers used here were etched chemically from a (100) silicon wafer by Kelvin Nanotechnology Ltd[3], are fabricated to be of a nominally identical design to previously studied samples [27–31] so that the longest dimension of the cantilever was aligned with the [110] crystal axis. At one end of each silicon cantilever, as shown in figure 1, there is a 10 mm by 5 mm by 0.5 mm thick 'clamping block' where the sample is clamped to the support structure in such a way as to reduce frictional energy losses at the clamp [32, 33].

Two nominally identical silicon cantilevers were studied, one of which was coated with the indium film, while the other remained uncoated as a reference sample. The flexing part of the cantilever is 34 mm long by 5 mm wide. Analysis of the resonant frequencies of an uncoated sample determined that the flexure was 54.6 μm thick [34]. Where possible, the mechanical dissipation of this reference cantilever was used in the analysis.

2.1. Film preparation

The indium film was applied to the silicon cantilever by means of thermal evaporation. The cantilever was mounted inside a high vacuum chamber and masked in such a way that only one of the polished faces of the 54.6 μm thick flexure component was coated. After the cantilever was removed from the sample mount, the indium film was determined, using an optical surface profiler, to be (530 ± 30) nm thick.

3. Experimental results

The temperature dependence of the mechanical dissipation of the coated sample was measured for a total of eight different resonant modes having frequencies between ~400 and 15000 Hz. The cantilever was mounted in a stainless steel clamp within the vacuum chamber of a temperature controlled cryostat[4]. The bending modes of the sample of angular frequency ω_0 were excited using an electrostatic actuator positioned a few millimetres below the cantilever. The dissipation $\phi(\omega_0)$ was found from a fit to the free exponential decay of the resonant motion [35]

$$A(t) = A_0 \mathrm{e}^{-\phi(\omega_0)\omega_0 t/2}. \tag{1}$$

The motion was sensed by illuminating the cantilever with a laser beam which then cast a shadow over a split photodiode sensor outside the cryostat.

Several measurement cycles were carried out during which the cantilever temperature was increased systematically from approximately 10–300 K, with the sample being removed and re-clamped between cycles. The temperature of the cantilever was recorded using a

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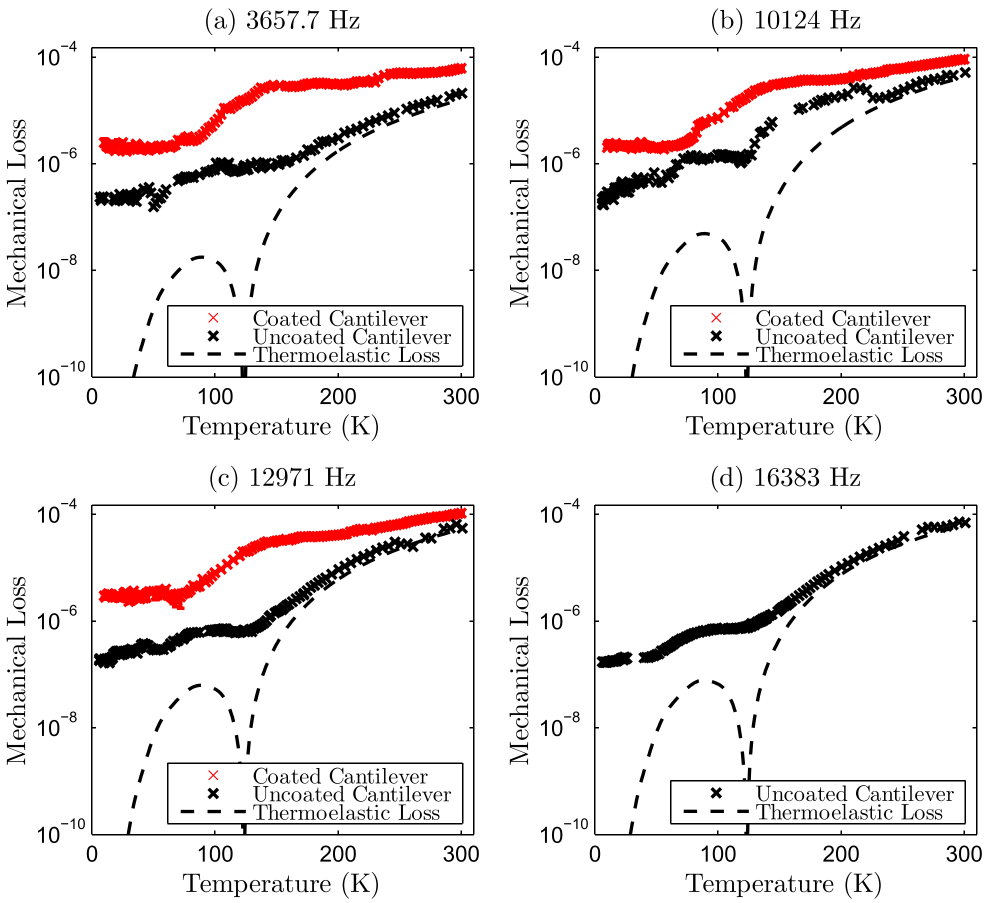

Figure 2. Measured mechanical loss of the 3658, 10124 and 12971 Hz resonant modes as a function of temperature of a 34 mm long by 5 mm wide by 54.6 μm thick silicon cantilever coated with a 530 ± 30 nm thick indium film (red). Also measured mechanical loss for four resonant modes of the silicon cantilever used as a nominally identical control (black), plotted together with the calculated thermoelastic loss of the substrate at each of the frequencies (dashed).

silicon-diode sensor (Lakeshore DT-670-SD) mounted on the clamp directly below the fixed end of the cantilever and Proportional-integral-derivative (PID) controlled with a Lakeshore Model 336 Cryogenic Temperature Controller which maintained the temperature to within 0.1 K. Repeated ring-down measurements at each temperature showed typically a variation in dissipation of less than 4% for each mode. The variation in the dissipation between repeated clamping was typically less than 10%. Full details of this experimental technique are discussed in [29]. Figures 2(a)–(c) show the results obtained for the silicon cantilever coated with the indium film for the resonant modes at 3658, 10124 and 12971 Hz.

3.1. Mechanical loss of thin films

The mechanical dissipation of the indium film can be calculated from the difference in the measured dissipation of the coated cantilever and an equivalent un-coated cantilever [35]:

Table 1. Mechanical properties of silicon [36].

Temperature (K)	Linear thermal expansion coefficient (K^{-1})	Spec. heat capacity ($J\ kg^{-1}K^{-1}$)	Thermal conductivity ($W\ m^{-1}K^{-1}$)
300	2.6×10^{-6}	705	140
20	-2.9×10^{-9}	3.41	4940

$$\phi(\omega_0)_{\text{coating}} = \frac{Y_s t_s}{3 Y_c t_c} (\phi(\omega_0)_{\text{coated}} - \phi(\omega_0)_{\text{substrate}}), \tag{2}$$

where ω_0 is the angular frequency of the resonant mode, $\phi(\omega_0)_{\text{coated}}$ is the loss factor of the coated cantilever, $\phi(\omega_0)_{\text{substrate}}$ is the loss factor of an equivalent 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 the thickness and Young's modulus of the coating. When a coating is thin in comparison to the substrate, the ratio of energy stored in the coating layer to the energy stored in the cantilever substrate is given by $\frac{3Y_c t_c}{Y_s t_s}$ [35].

Cryogenic mechanical loss measurements were made of a nominally identical silicon un-coated cantilever from the same batch of cantilevers for four resonant modes. Three of these resonant modes coincided in frequency with those measured on the indium coated cantilever, as shown in figure 2. This provided some suitable control data to calculate the loss of the indium film. Additional resonant modes at 394, 1124, 2211, 5641 and 7615 Hz were measured for the coated sample. However, as discussed later, it is possible to use the losses measured on the 16383 Hz resonant mode of the un-coated cantilever as a source of values in equation (2) to calculate $\phi(\omega_0)_{\text{coating}}$ for these resonant modes.

3.2. Young's modulus of silicon and indium

The Young's modulus of ⟨110⟩ silicon was taken to be 166 GPa [36], and since it varies by less than a few percent over the temperature range studied [36, 37] it was therefore assumed to be constant in this analysis. However, the Young's modulus of indium does vary with temperature from 12.61 GPa at 300 K, 18.36 GPa at 80 K to 19.56 GPa at 5 K [38]. A polynomial interpolation was then used to estimate the Young's modulus of indium between 10 and 300 K.

3.3. Thermoelastic loss

When a body is at rest, deformation thermal expansion in the material can still result from localized statistical temperature fluctuations. The resulting thermoelastic dissipation in the flexure is a function of the thermal expansion coefficient of the material, α, and other properties [39, 40], so that it can be calculated using [40, 41]:

$$\phi(\omega) = \frac{Y\alpha^2 T}{\rho C} \frac{\omega\tau}{1 + \omega^2\tau^2}, \tag{3}$$

where Y is Young's modulus, ρ is density, C is specific heat capacity and τ is the relaxation time. This relaxation time, the time to return to a thermal equilibrium, is related to the time taken for heat to flow across the cantilever and for a rectangular cross-section, of thickness t, and can be shown to be

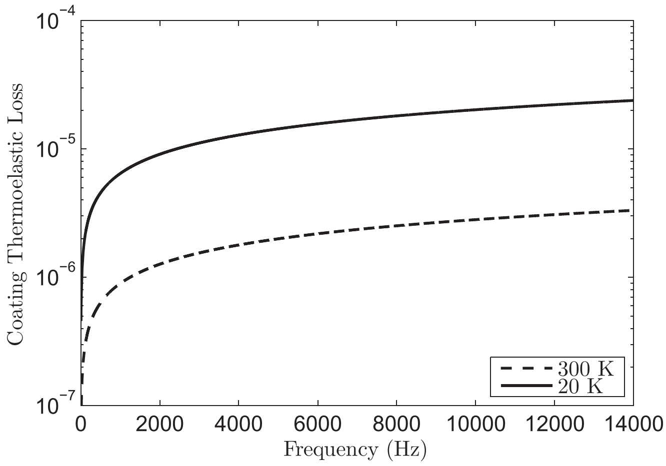

Figure 3. Estimate of the coating thermoelastic loss for the indium coating at 300 and 20 K.

$$\tau = \frac{\rho C t^2}{\pi^2 \kappa}, \tag{4}$$

where κ is thermal conductivity of the material [42]. The mechanical properties of silicon near room temperature and at low temperature are detailed in table 1.

Figure 2 shows the calculated thermoelastic loss as a function of temperature for four resonant modes of a 54.6 μm thick silicon cantilever along with the measured mechanical loss of the cantilever. It is clear that above 200 K the mechanical loss of this cantilever is dominated by thermoelastic loss, while at lower temperatures the level of the mechanical loss has been shown to depend on the surface quality [27].

3.4. Coating thermoelastic loss

A further source of loss to be considered when measuring the dissipation in a coated sample originates from the differing thermo-mechanical properties of the coating and substrate materials. Fejer *et al* [43] derived the coating thermoelastic loss for a uniform thin film. The magnitude of this dissipation depends strongly on the difference between the properties of the substrate of and the coating. The mechanical properties of indium at 300 and 20 K used to estimate the coating thermoelastic loss, are given in table 2.

A nominal operating temperature for cryogenic gravitational wave detectors is approximately 20 K [3]. It is, therefore, of interest to estimate the loss of the film at that temperature. The magnitude of the coating thermoelastic loss for the 530 nm thick layer on a silicon substrate was calculated, using equations (5) and (6) of Fejer *et al* [43] at both room temperature and at 20 K, as shown in figure 3.

The level of coating thermoelastic loss at room temperature is calculated to be of the order 10^{-6}, which is several orders of magnitude below the calculated loss of the indium film and consequently understood to have little or no effect on the loss. At 20 K the level of coating thermoelastic loss is approximately 50 times lower than the levels of indium as calculated for figures 5 and 7 and thus is still far from having any significant effect on the loss of the indium film.

Table 2. Mechanical properties of indium at 300 and 20 K used in the calculation of the coating thermoelastic loss [44].

Temperature (K)	Linear thermal expansion coefficient (K^{-1})	Spec. heat capacity ($J\ kg^{-1}K^{-1}$)	Thermal conductivity ($W\ m^{-1}K^{-1}$)
300	3.2×10^{-5}	233	81.8
20	0.7×10^{-5}	60.8	180

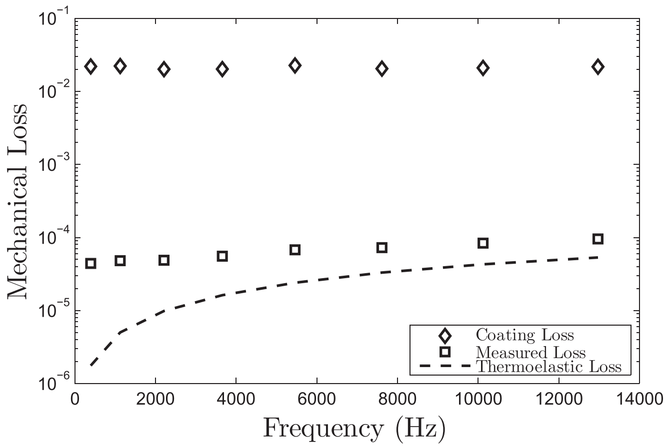

Figure 4. Measured mechanical loss and calculated coating loss of eight resonant modes at room temperature of the silicon cantilever coated with a thermally evaporated indium layer, plotted together with the predicted levels of thermoelastic loss as a function of frequency at room temperature.

3.5. Mechanical loss at room temperature

Mechanical loss measurements for eight resonant modes of the indium coated silicon cantilever, ranging between ~400 and 15,000 Hz, were measured at room temperature before the cryostat was cooled. They are presented in figure 4 and compared to the predicted levels of thermoelastic loss for a 54.6 μm thick cantilever.

Figure 2 showed that at room temperature the levels of substrate loss are dominated by the levels of thermoelastic loss and thus can provide an accurate estimate of the substrate loss. The coating loss was calculated from the measured data using equation (2).

From figure 4 it is clear that the levels of coating loss for the evaporated indium film are at a similar level across the frequency range measured at room temperature. The average coating loss was found to be 0.021 ± 0.001 which is broadly consistent with the loss measured for a hydroxide catalysis bond [21]. It is, however, higher than the ~2×10^{-3} loss measured by Liu *et al* [26] with this increase possibly due to impurities introduced during thermal evaporation. The most likely impurities in this process are metallic contaminants from the crucible used to hold the indium prior to evaporation. It is also highly likely that the surface of the indium oxidized on exposure to the air, and this oxide layer may also contribute to the higher loss with respect to Liu's results.

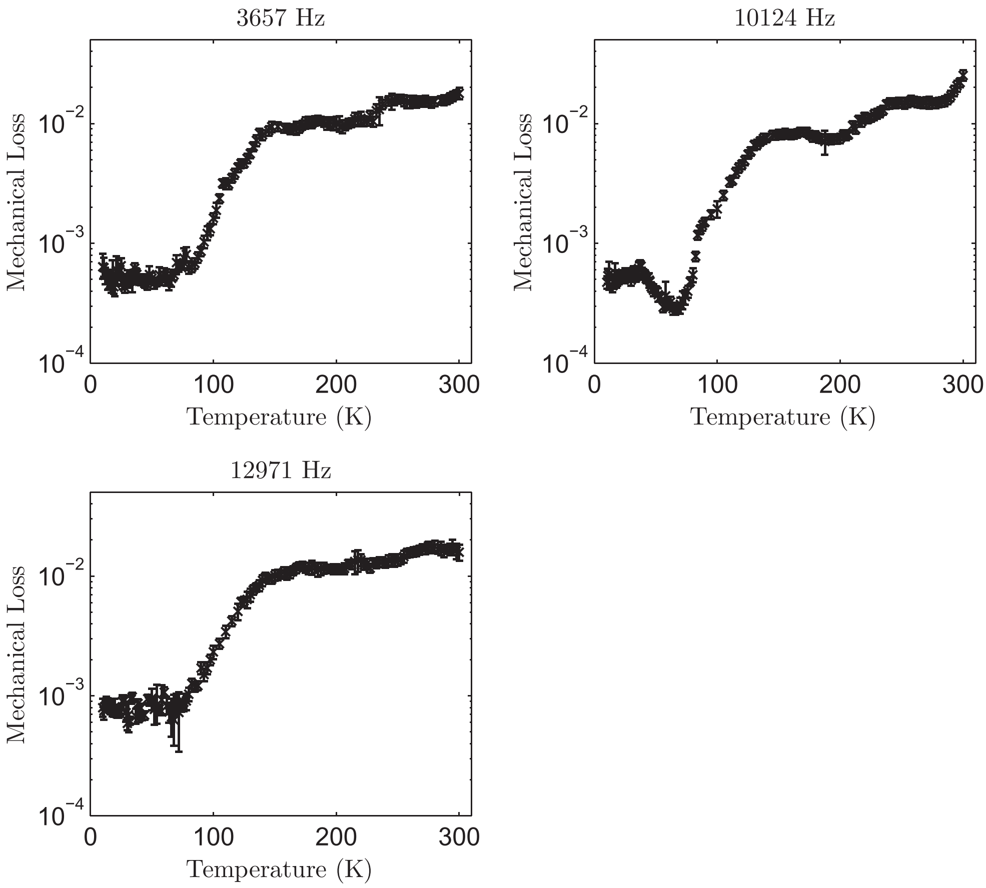

Figure 5. Mechanical loss of the indium coating as a function of temperature, for the resonant modes where there was control data at matching frequencies.

4. Mechanical loss at low temperatures

The mechanical losses of eight resonant modes were measured in a temperature range from 10–300 K. The temperature was increased using a PID controller and left to stabilize before each temperature step was measured. Figure 2 summarizes the mechanical loss values measured for the resonant modes at 3657, 10124 and 12971 Hz. Taking into account the temperature dependence of the Young's modulus of indium, as discussed earlier, a similar analysis was undertaken to calculate the loss of the indium film for each temperature step between 10 and 300 K for these resonant modes using the control data measured for the same modes, at approximately the same frequencies, of an un-coated 'control' sample. The mechanical loss of the indium film as a function of temperature for these resonant modes is shown in figure 5.

However, for the remaining five resonant modes at 394, 1124, 2211, 5641 and 7615 Hz there was no un-coated cantilever loss data that matched in exact frequency to these resonant modes. It is still possible to calculate an upper limit for the loss of the indium film at each of those resonant modes. Firstly, the mechanical loss of the indium layer was calculated using the predicted level of thermoelastic loss at that frequency for a 54.6 μm micron thick silicon

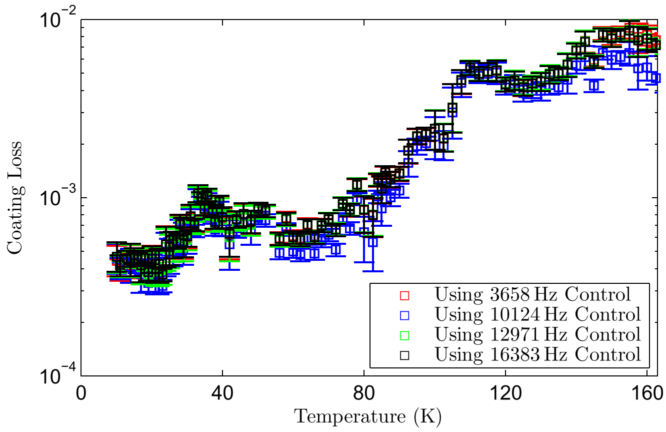

Figure 6. Comparison of the calculated levels of mechanical loss of the indium coating for the 394 Hz resonant mode calculated using the control data measured on the 3658 Hz (red), 10124 Hz (blue), 12971 Hz (green) and 16385 Hz (black) resonant modes up to 160 K.

cantilever for the substrate loss. The resulting coating loss is indicated by the dashed line in figure 7. Using the thermoelastic loss of the cantilever in this way provides a good approximation to the loss of an un-coated cantilever at temperatures above 120 K, where thermoelastic damping begins to become the dominant source of loss in the un-coated cantilever. At lower temperatures, where the thermo-elastic loss becomes orders of magnitude lower than the loss of the cantilever (see figure 2), this approximation effectively treats the un-coated cantilever as being lossless, and thus provides a good upper limit for the coating loss.

However, from figure 2 it is clear that the substrate loss would be expected to be significantly larger than the thermoelastic loss at low temperatures. Thus, to refine the upper limit calculated above, an analysis similar to that undertaken for the 3657, 10124 and 12971 Hz resonant modes was made for the additional five resonant modes. For each of these modes, the coating loss was calculated using the un-coated control data for each of the four measured resonances in figure 2. Figure 6 shows, as an example, the coating loss results obtained for the 394 Hz mode of the coated sample. It is clear that using the different sets of control data makes less than 5% difference to the coating loss below 150 K to the calculated coating loss on this, and the remaining modes. Therefore, the mechanical loss of the indium was calculated for the remaining resonant modes a second time, below 150 K, using the un-coated data measured for the 16385 Hz mode, in figure 2(d), as a more realistic upper-limit approximation of the loss of the cantilever substrate at low temperature, as shown in figure 7.

5. Discussion

Figures 5 and 7 show that the loss of the coating has a broadly consistent trend across all the resonant modes. There is evidence of a plateau in the loss between about 40 and 50 K with over an order of magnitude reduction in loss at low temperature compared to room temperature. Figure 5 indicates there is a broad dissipation peak around 150 K possibly indicating a thermally activated relaxation effect due to stress induced motion of dislocations, or mobile point defects in metals [45].

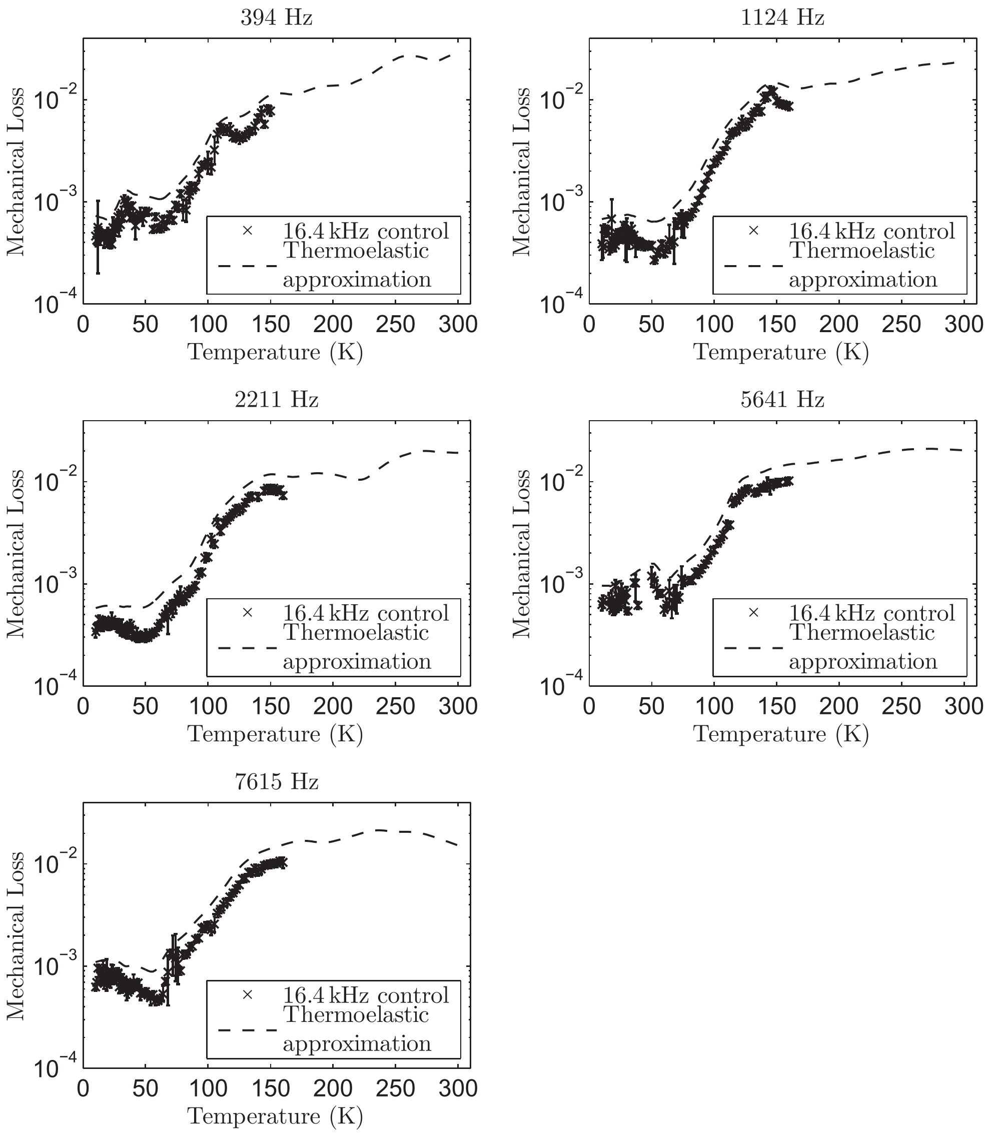

Figure 7. Mechanical loss of the indium coating as a function of temperature, for the resonant modes where there was no matching control data, calculated firstly using thermoelastic loss and secondly using the data measured on a 16385 Hz resonant mode up to 150 K.

Table 3 shows that the level of mechanical loss across the resonant modes measured on the coated cantilever range between 4 and 8×10^{-4} at 20 K. The loss of the coating at 295 K for each of these resonant frequencies is shown to highlight the reduction in loss at 20 K. Where possible $\phi(\omega_0)_{\text{coating}}$ was calculated using data from the matching resonant mode of the un-coated cantilever, and the entries in italics indicate the losses calculated using the loss values measured for the 16385 Hz mode of an un-coated cantilever where there was no matching control data. As shown in figure 8, the results show similar trends to the mechanical losses measured by Liu *et al* [26]. Although both sets of results are, in general, in close

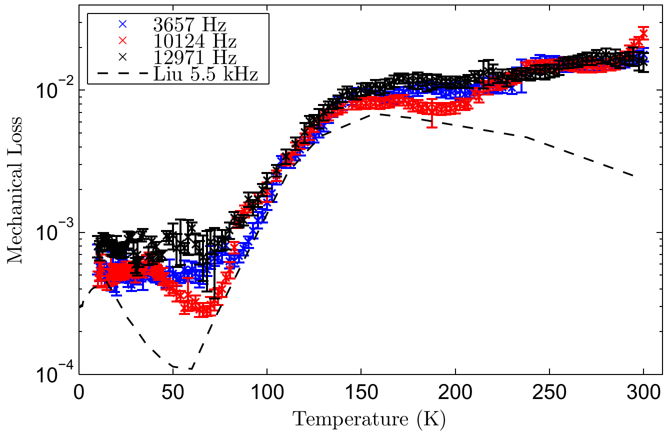

Figure 8. Mechanical loss of the indium coating as a function of temperature, for the resonant modes where there was matching control data compared to the levels of loss estimated by Liu [26].

Table 3. Summary of $\phi(\omega_0)_{\mathrm{coating}}$ of the indium film at both 295 and at 20 K. Italics indicate that the coating loss was calculated using the 16385 Hz upper-limit approximation.

Frequency (Hz)	$\phi(\omega_0)_{\mathrm{coating}}$ at 295 K ($\times$ 10^{-2})	$\phi(\omega_0)_{\mathrm{coating}}$ at 20 K ($\times$ 10^{-4})
394	2.2 ± 0.3	*4.8 ± 0.8*
1124	2.2 ± 0.2	*4.1 ± 0.7*
2211	2.0 ± 0.2	*4.1 ± 0.3*
3657	2.0 ± 0.2	4.6 ± 1.0
5641	2.3 ± 0.2	*5.9 ± 1.1*
7615	2.1 ± 0.2	*7.4 ± 1.1*
10124	2.1 ± 0.1	5.1 ± 0.5
12971	2.2 ± 0.1	8.1 ± 1.2

agreement, at room temperature and at temperatures around 50 K, Liu's losses were significantly lower than the losses measured here. It is possible that this is due to the different deposition techniques used. The film measured by Liu was deposited by e-beam evaporation, and would thus be expected to have fewer impurities than the thermally evaporated film studied here. It is also possible that there were differences in the degree of oxidation of the surface of the indium films, which could potentially significantly alter the measured loss.

The resulting losses at cryogenic temperature range from a factor of ~25 to 50 times lower than they were on average at room temperature, indicating indium to be of potential interest for use in a detector at cryogenic temperatures. Further measurements with an indium bond constrained between substrates are consequently of extreme interest in order to estimate better the levels of thermal noise which would be contributed from an indium joint.

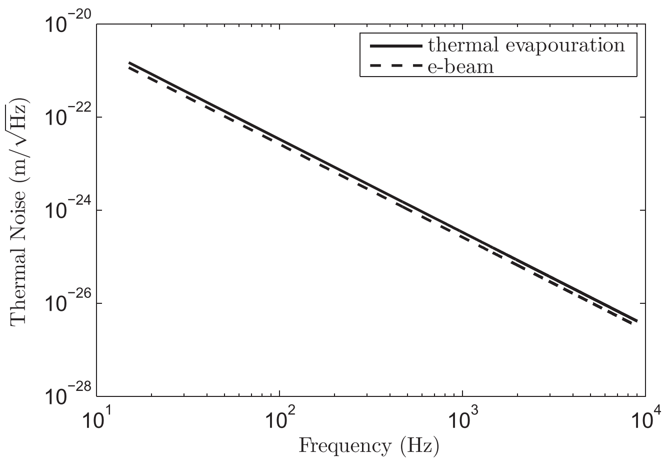

Figure 9. Thermal noise of a 340 mm diameter and 200 mm thick sapphire mirror, with 95 mm wide flats on diametrically opposite sides of the cylindrical face onto which two sapphire ears are jointed using a thermally evaporated 530 nm thick layer of indium and an e-beam evaporated film of the same thickness.

6. Thermal noise contributed by an indium layer between interfaces on a typical test mass at 20 K

The thermal noise contribution of a thin indium layer located between small sapphire interface pieces (ears) between the fibre suspension elements and the mirror of a gravitational wave detector can be estimated using an analysis similar to Cunningham *et al* [21]. Following Levin's approach, the thermal noise of a mirror can be evaluated by calculating the average power dissipated, $W_{\rm diss}$, when a notional oscillatory force of peak magnitude F_0 and having the same spatial profile as the interferometer laser beam, acts upon the face of the mirror. Levin shows that the power spectral density of the thermally induced displacement on the front face of the mirror is given by

$$S_x(f) = \frac{2k_B T W_{\rm diss}}{\pi^2 f^2 F_0^2}. \tag{5}$$

It can be shown that the loss of the material in the system $\phi(x, y, z, f)$ at frequency f is related to the power dissipated

$$W_{\rm diss} = 2\pi f \int_{\rm vol} \epsilon(x, y, z)\phi(x, y, z)\mathrm{d}V, \tag{6}$$

where ϵ is the energy density of the maximal deformation of the test mass under the applied notional pressure. As an example, we consider the case of a sapphire mass with dimensions similar to that of an Advanced LIGO optic; 340 mm diameter and 200 mm thickness, with 95 mm wide flats on diametrically opposite sides of the cylindrical face. The ears for attaching sapphire suspension elements are jointed on each of the flats.

Using Levin's approach, with the loss values reported here at 20 K for an indium film together with the relevant parameters, it is possible to calculate, using finite element analysis, the overall thermal noise contribution at 100 Hz of an indium bond layer on a sapphire test mass for both thermally and e-beam evaporated films, as shown in figure 9. For a 55 mm beam radius, as used in Advanced LIGO, with a 530 nm thick indium layer it was established that, for a single test mass, the thermal noise associated with a thermally evaporated indium layer between the ears would be $(3.3 \pm 0.3) \times 10^{-23}\,\mathrm{m}/\sqrt{\mathrm{Hz}}$ at 20 K. This value is approximately 16 times lower than the estimated thermal noise associated with a silicate bond between fused silica components at room temperature [21] and almost a factor of a hundred lower than the total noise budget, per mirror, for the KAGRA design [3], and could be marginally improved through the use of an e-beam evaporated film.

7. Conclusion

This paper has presented new mechanical loss values for a thermally evaporated indium film and shown that the loss associated with it is comparable to the loss of a hydroxide-catalysis bond at room temperature. It has shown that the loss of the indium film reduces significantly at low temperature, making it of extreme interest for the KAGRA detector. It should be noted that, at the 20 K operation temperature of KAGRA, the results presented here are very close to Liu's results. For operation at temperatures between 20 and 60 K, an e-beam film appears likely to be a good candidate for providing lower thermal noise. Calculations have shown that the thermal noise for an indium layer thermally evaporated between two typical components of an advanced detector is $(3.3 \pm 0.3) \times 10^{-23}\,\mathrm{m}/\sqrt{\mathrm{Hz}}$, which is well below the requirements for the KAGRA detector.

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