Mechanical Loss Angle Measurement for Stressed thin Film Using Cantilever Ring-Down Method

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Mechanical loss of the coating materials, and hence thermal noise from the mirror coatings, is a limiting factor for the sensitivity of the laser interferometer gravitational waves detector at its most sensitive frequency range. Mechanical loss of the thin films are often measured using the cantilever ring-down method. But when the thin film is under stress, the regular ring-down method gives incorrect results. We report a method to obtain the mechanical loss of stressed thin film using the cantilever ring-down technique. A proof-of-concept example is given to demonstrate and verify our method. The method can also be applied to obtain the mechanical loss angle of a rough interface; an example showed that loss angle of the interface between silicon nitride film deposited by plasma enhanced chemical vapor deposition method and silicon substrate is highly frequency dependent.

Keywords: mechanical loss angle, laser interferometer gravitational waves detector, mirror coatings, stressed thin film.

1. Introduction

Sensitivity of the current laser interference gravitational waves detector at its most sensitive frequency region ~100 Hz is limited by the thermal noise of the mirror coatings. Thermal noise is related to the mechanical loss angle through the fluctuation-dissipation theorem. Therefore, studying the mechanical loss of the coating materials is crucial for the development of better coatings of the detector. Cantilever ring-down is a common method for measuring the mechanical loss of coatings. A cantilever, usually silicon, with a thicker clamping pad is fabricated and the coating is deposited on one side of the thinner cantilever. The cantilever is clamped on the pad and excited under vacuum at its resonant frequencies. The decay time of the free damping is recorded and the loss angle is obtained through Eq.1. The loss angle of the film can be deduced from the difference between coated and un-coated cantilevers. The dimensions of the cantilever are designed such that the resonant frequencies fall in the range of interest, i.e. from several tens to several thousand Hz. The thickness of the cantilever is usually about 100 μm or less. The pad needs to be thick enough to avoid energy coupling between the clamp and the cantilever during the ring-down measurement. To fabricate the cantilever one starts with a double-side polished thick silicon wafer, using a lithographic method and KOH etching on one-side to form the cantilever of the desired thickness. The resulting cantilever substrate, shown in Fig. 1(a), is polished on one side and roughened by the KOH etching on the other, the roughness was typically 7.7 nm as was measured by atomic force microscopy (AFM). The coating is then deposited on the polished side of the cantilever for the ring-down measurement.

When the coating is stressed, the coated cantilever warps as shown in Fig. 1(b), which may lead to inaccurate ring-down measurement. To eliminate the warping, we propose coating the cantilever on both sides to balance the stress as shown in Fig. 1(c). However, the film on each side is different in the sense that one film is deposited on the smooth surface and the other film is deposited on the roughened surface. We developed a method to separate the loss angle of the film on the smooth surface and the other film is deposited on the roughened surface. We developed a method to separate the loss angle of the film on the smooth surface and the other film is deposited on the roughened surface. We developed a method to separate the loss angle of the rough interface between the film and the substrate. Examples are given to demonstrate the validity of our methods.

2. Sample Configuration

Two types of cantilever samples were prepared. Type I is a double-side coated cantilever which is one-side roughened while the other side is left polished as shown in Fig. 1(a) and (c). Type II is a double-side coated cantilever which is roughened on both sides of the substrate as shown in Fig. 2(a) and (c). The roughness on both sides have to be created under exactly the same condition such that they are identical in roughness. After the stressed film is deposited on one side at an elevated temperature, due to differential

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thermal expansion, the cantilever warps on cooling down as shown in Fig. 1(b) and Fig. 2(b). When coating the film on the second side, the cantilever is brought up to the same elevated temperature and the differential thermal expansion stress is released so that the cantilever becomes flattened. On cooling down after the second coating, the cantilever remains flat due to stress balance. The resultant sample configurations are shown in Fig. 1(c) and 2(c) and referred to as type I and type II.

![Figure 2. Curvature variation of the samples with double-side coatings on both-side roughened cantilever.](image)

### 3. Loss Angle Calculation

The loss angle \( \phi \) is defined as the ratio of the oscillation energy dissipation per cycle to the elastic stored energy

\[
Q^{-1} = \phi = \frac{P}{\omega E} = \frac{2}{\omega \tau}
\]

where \( P \) is the power dissipation, \( E \) is the stored energy, \( \omega \) is the angular frequency and \( \tau \) is the decay time for the resonant mode at \( \omega \).

The loss angles of the one-side roughened bare cantilever, \( \phi_s \), double-side roughened bare cantilever \( \phi_s' \), type I cantilever \( \phi_r \), and type II cantilever \( \phi_{r'} \) are shown in Table 1. Because the roughness of the interface is much less than the thickness of the film and the thickness of the substrate is much larger than that of the film, we can approximate \( E_s \approx E_s' \approx E_r' \) and \( E_s \approx E_r > E_r' \). Under these assumptions, the loss angles can be approximated as shown in Table 1.

The total power dissipation in the sample is the sum of the power dissipation of all components. It is simple to show from Table 1, the loss angle of the film that is coated on the polished side, \( \phi_f \), is

\[
\phi_f = \frac{1}{\omega} \frac{P_f}{E_f} \approx \frac{E_s}{E_f} \left[ (\phi_i - \phi_s) - \frac{1}{2} (\phi_{ii} - \phi_{s'}) \right]
\]

Notice that the loss angles in the parentheses can be obtained by direct ring-down measurement on the four samples in Table 1.

When the cantilever is excited with the bending modes, the energy ratio can be approximated as \(^{9,10}\):

\[
\frac{E_i}{E_f} = \frac{Y_s t_s}{3Y_f t_f}
\]

One would need to prepare one-side roughened bare cantilever \( s \), double-side roughened bare cantilever \( s' \), double-side coated cantilever type I and double-side coated cantilever type II samples and measure the loss angles, \( \phi_s, \phi_s', \phi_{r}, \phi_{r'} \), to extract \( \psi \). The Young's modulus and shear modulus of the silicon substrate, \( Y_s \) and \( G_s \), can be obtained from the literature\(^{12}\). The Young's modulus of the films, \( Y_f \), can be measured by using instruments such as nano-indenter. If the films are amorphous, i.e. homogeneous and isotropic, shear modulus of the films, \( G_f \), can be obtained from the Young's modulus through the following equation\(^3\), assuming that the Poisson ratio of the films, \( \nu_f \), is known:

\[
G_f = \frac{Y_f}{2(1+\nu_f)}
\]

The loss angle of the rough interface can also be obtained as follows. The film \( f' \) which is deposited on the rough surface can be decomposed, in terms of power dissipation and energy stored, into two parts as shown in Fig. 3. One part of the film which is smooth on both surfaces and the other part is the rough interface. Following the method of derivation of Eq. 2, the loss angle of the interface, \( \phi_r \), can be obtained as:

\[
\phi_r = \frac{1}{\omega} \frac{P_r}{E_r} \approx \frac{E_s}{E_f} (\phi_r - \phi_{f'})
\]

where \( \phi_{f'} \) is the loss angle of the one-side-rough film. When the cantilever is excited with a bending mode, the energy ratio is approximated as:

\[
\frac{E_{r'}}{E_r} = \frac{Y_s t_s}{3Y_f t_f}
\]

When the cantilever is excited at the torsional mode, the Young's moduli \( Y_r, Y_f \) should be replaced by shear moduli \( G_r, G_f \).

\( E_s \) is the energy stored in the rough interface, \( Y_r \) is the effective Young's modulus of the interface, we assume \( Y_r \) can be taken as the average of the Young's modulus of the film and the substrate. The same assumption holds for the shear modulus of the rough interface \( G_r \), \( t_s \) is the effective thickness of the interface that can be taken as the roughness of the surface.

![Figure 3. The one-side-rough film \( f' \) can be decomposed, in terms of power dissipation and energy stored, into the smooth film \( f \) and the rough interface \( r \).](image)
Table 1. Mechanical loss of different samples.

| Sample Description | Mechanical Loss |
|--------------------|----------------|
| One-side roughened bare cantilever | $\varphi_s = \frac{1}{\omega} \frac{P_s}{E_s}$ |
| Double-side roughened bare cantilever | $\varphi_s' = \frac{1}{\omega} \frac{P_s'}{E_s'} \approx \frac{1}{\omega} \frac{P_s}{E_s}$ |
| Film on smooth surface | $\varphi_I = \frac{1}{\omega} \frac{P_s + P_f + P_f'}{E_s + E_f + E_f'} \approx \frac{1}{\omega} \frac{P_s + P_f + P_f'}{E_s}$ |
| Film on rough surface | $\varphi_{II} = \frac{1}{\omega} \frac{P_s + P_f + P_f'}{E_s + E_f + E_f'} \approx \frac{1}{\omega} \frac{P_s + P_f + P_f'}{E_s}$ |

$s$: One-side roughened bare cantilever
$s'$: Double-side roughened bare cantilever
$f$: Film on smooth surface
$f'$: Film on rough surface

4. Proof of Concept

4.1 Loss angle of the stressed silicon nitride film

The photo-lithographic method was used followed by KOH etching to fabricate one-side and two-side roughened cantilevers. The surface roughness of the commercial double-side polished silicon wafer is usually < 0.5 nm, and the KOH etching produced surface roughness of 7.7 nm. Plasma enhanced chemical vapor deposition (PECVD) was used to deposit the silicon nitride film on the cantilevers. The film was amorphous as was revealed by its electron diffraction pattern. The thickness of the film was 199 ± 1 nm. The composition of the film was SiN$_{0.65}$ measured by X-ray photoelectron spectroscopy (XPS). The Young's modulus was 131.6 ± 4.8 GPa, measured by nano-indenteter. The Poisson ratio was taken as 0.25$^{15}$. The Young's modulus and shear modulus of the silicon substrate were taken as 169 GPa and 79.6 GPa$^{12}$, respectively. The one-side coated cantilever warped with the coating on the concave face indicating tensile stress of the film. The stress was 256.7 ± 6.6 MPa, measured by a curvature meter and calculated by using the Stoney equation$^{16}$. The value and the rms error were obtained from five measurements along five diameters on a coated 4" silicon wafer. The double-side coated cantilever was flat, with the radius of curvatures > 10$^3$ m before and after double-side coating, indicating that good balance of the stresses was achieved. The loss angles, $\varphi_s$, $\varphi_s'$, $\varphi_f$, and $\varphi_{II}$, were measured and the loss angle of the film $\varphi_f$ was calculated according to the method described. Fig. 4 shows the loss angle of the film, in the black square, for five modes of the cantilever.

![Figure 4. The loss angle of SiN$_{0.65}$ deduced by different methods.](image)

In order to verify our method, we developed a technique to fabricate a double-side polished cantilever with clamping pad from a silicon-on-insulator (SOI) wafer$^{17}$. The double-side polished cantilever had the same dimensions as the regular silicon cantilevers and the surface roughness was < 0.5 nm for both sides. We then deposited the same films on both sides of the cantilever and measured the loss angle of the film using the ring-down method. The result is shown in Fig. 4 as the red dots. The loss angles of the film on regular silicon cantilevers deduced using our method are nearly the same, within the error bars, of the measurement as that form the double-side polished cantilever. However, the SOI wafer is much more expensive than the regular silicon wafer and it is more complicated to fabricate into the shape of the cantilever that routine usage for our purpose is discouraged.
4.2 Loss angle of the rough interface

The film for this example was the same as the previous example except that the composition was SiN$_{0.87}$. The thickness of the film was $219 \pm 1$ nm. The Young's modulus was $138.0 \pm 4.7$ GPa. The tensile stress was $423.4 \pm 5.9$ MPa. Type I and II samples were fabricated and all the relevant loss angle were measured and calculated. The loss angle of the rough interface, $\phi_r$, can then be calculated according to Eq.6. The result is shown in Fig. 5. The roughness of the interface created by KOH etching for this sample was 7.7 nm as was measured by using an atomic force microscope (AFM). Fig. 5 hints that the mechanical loss of the rough interface is highly frequency dependent. However, the full interpretation of Fig. 5 is outwith the scope of this paper.

5. Conclusion

We proposed a method to obtain the mechanical loss angle of a stressed thin film which is coated on a cantilever with surface roughness. We showed that the method is effective by demonstration through an example. Our method can also be applied to obtain the effective mechanical loss angle of a rough interface between a film and a substrate. The measurements show that the mechanical loss angle of the rough interface appears to be highly frequency dependent.

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