A Microcantilever Platform for Measuring Internal Friction in Thin Films Using Thermoelastic Damping for Calibration

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Abstract—Measuring structural damping due to internal friction (IF) in deposited thin films can generate valuable insights into the effects of size and confinement on the mechanisms of anelasticity and provide useful guidelines for the design of high-Q micro/nanomechanical resonators used in microelectromechanical systems for sensing, communications, and vibration energy harvesting. However, accurate measurement of IF has been a longstanding challenge because of the difficulty associated with calibrating the background damping. In this paper, we present an approach based on a silicon microcantilever platform that resolves these difficulties. This approach is built upon the ability to operate silicon microcantilevers at the fundamental limits of dissipation established by thermoelastic damping (TED) and the ability to compute TED in metal-coated silicon beams without using any free parameters. Using this method, we present the first accurate measurements of IF at room temperature in films of aluminum, silver, and gold with thickness ranging from 50 to 500 nm. Gold films dissipate less energy than either aluminum or silver at the frequencies measured in this paper. In all cases, IF is dominated by defect-induced processes occurring within the bulk of these films and not at the silicon/film interface or at the free surface of the metals. [2010-0289]

Index Terms—Damping, internal friction (IF), microcantilever platform, thin films.

I. INTRODUCTION

THE PRIMARY objective of this paper is to describe a technique for accurate measurement of structural damping due to internal friction (IF) in deposited thin films. Such measurements can lead to useful guidelines for improving the performance of an important class of resonant microelectromechanical systems (MEMS) and enable fundamental studies of the mechanical behavior of thin-film materials. Some examples are cited below by way of illustration.

Micromechanical and nanomechanical resonators are the critical components of MEMS used for sensing (resonant sensors to detect physical, chemical, and biological signals), communications (filters, timers, and frequency references), and energy harvesting by converting ambient mechanical motion into electrical power. All these applications require resonators with very high mechanical quality factors of resonance (10⁴ < Q < 10⁶) to enhance performance and functionality (see, for example, [1]–[6]). Alternately, this requirement may be stated in terms of the logarithmic decrement δ of the resonator. For the low values of dissipation under consideration

\[ \delta = \frac{\pi Q}{W_{\text{max}}} \]  

where ΔW is the energy dissipated per cycle of vibration and W_{\text{max}} is the maximum elastic energy stored in the resonator [5]. We will use δ as the dimensionless measure of damping in this paper.

Significant progress has been made in the design of vacuum-operated monolithic ceramic resonators with very low damping (10⁻⁶ < δ < 10⁻⁴). Resonators fabricated using single-crystal silicon, polysilicon, silicon nitride, and silicon germanium can approach the fundamental limits of dissipation established by thermoelastic damping (TED) [7]–[14]. However, for many applications, it is common practice to coat these ceramic beams with thin metallic films to increase electrical conductivity and optical reflectivity and to alter surface chemistry. Experience with several devices suggests that these coatings can increase damping; in some cases, relatively thin metallic films have been found to degrade the quality factor of ceramic beams by an order of magnitude [1], [15]–[18]. A systematic study of the effects of thickness, frequency, temperature, microstructure, and alloying additions on structural damping in thin metallic films can provide useful guidelines for selecting materials, dimensions, processes, and operating conditions to optimize the design of layered resonators and attain high quality factors. No such study has yet appeared in the literature, and the work presented in this paper is a first step toward that goal.

In parallel, careful measurements of IF can provide valuable insight into the effects of size and confinement on the mechanical behavior of thin-film materials. In this context, IF refers to energy dissipation and damping that is related to the density, distribution, and mobility of crystallographic defects (vacancies, substitutional and interstitial impurities, surface defects, dislocations, grain boundaries, and phase boundaries). The analysis of the frequency dependence of IF is called mechanical spectroscopy, and this technique has been extensively used to characterize bulk solids [5], [6]. However, in stark contrast...
to the vast and growing literature on most other aspects of their mechanical behavior [19], very little is known about IF in thin films. This gap in knowledge can be filled by developing techniques for mechanical spectroscopy of deposited thin films.

The earliest attempts to measure IF in thin films can be traced back to the 1960s. In 1963, Weiss and Smith suggested the use of a composite structure consisting of a thin film deposited on a low-loss quartz ribbon to measure IF in the film [20]. Building upon this idea, Berry and Pritchett published a series of influential papers in which they described a vibrating reed apparatus (consisting of thin films deposited on miniaturized cantilevers) and established a protocol for measurement and analysis of IF [21]–[25]. The essential features of this protocol, which has now become the standard in this field, can be summarized in a sequence of four steps. The first step is to return to the important point later in this paper.

The third step is to measure the logarithmic decrement of the bare substrate \( \delta_s \) by recording the decay of the amplitude of free vibrations. All measurements are performed at sufficiently low pressures to ensure that viscous losses due to air damping or squeezed film damping are negligible. The fourth step is to deposit the film of interest and measure the damping of the composite film–substrate system \( \delta_c \).

At this point, it is necessary to confront the difficult question of extracting the IF in the film from the damping measured in the substrate and bilayer. To this end, Berry and Pritchett suggested a layer-by-layer partitioning of the dissipated energy so that [21]

\[
\delta_c = \frac{\Delta W_c}{2W_{c,max}} = \frac{\Delta W_s}{2W_{s,max}} + \frac{\Delta W_f}{2W_{c,max}}. \tag{2}
\]

In this expression and in what follows, the subscripts \( c \), \( s \), and \( f \) denote the composite, substrate, and film, respectively. Equation (2) assumes that the film is perfectly attached to the substrate. In the limiting geometry of a thin film on a thick substrate with \( h_f \leq 0.01 h_s \), \( W_{c,max} \approx W_{s,max} \) [21]; therefore, (2) can be expressed as

\[
\delta_c = \frac{\Delta W_s}{2W_{s,max}} + \frac{\Delta W_f}{2W_{s,max}}. \tag{3}
\]

The first term on the right-hand side can be identified as the logarithmic decrement of the bare substrate. The second term can be suitably normalized to obtain the logarithmic decrement of the film if it were to be measured independently under the same conditions [21]. Thus,

\[
\delta_c = \delta_s + \frac{W_{f,max}}{W_{s,max}} \frac{\Delta W_f}{2W_{f,max}} = \delta_s + \frac{W_{f,max}}{W_{s,max}} \delta_f
\]

\[= \delta_s + \left( \frac{3E_f h_f}{E_s h_s} \right) \delta_f \tag{4}
\]

where \( E \) is the Young’s modulus. The term in the parenthesis is the ratio of the maximum stored elastic energy in the film and substrate. This ratio can be expressed in terms of the elastic modulus and thickness of the film and substrate by a standard analysis of the quasi-static deformation of an Euler–Bernoulli beam [21].

Equation (4) has been used in numerous studies to extract the value of \( \delta_f \) from the measured values of \( \delta_s \) and \( \delta_c \) [21]–[36]. Furthermore, \( \delta_f \) is commonly identified as the IF caused by the motion of crystallographic defects in the film. This identification, unfortunately, is not accurate. Each of \( \delta_s \), \( \delta_f \), and \( \delta_c \) contains contributions from several mechanisms of dissipation, and an error of unknown magnitude is incurred if \( \delta_f \) is taken as the estimate of IF. This feature of the standard protocol has been noted before. In 1992, Bohn and Su suggested that (4) must be modified to incorporate an additional term to account for these other sources of dissipation, which they termed background damping (BG) [26]; hence,

\[
\delta_c = \frac{\Delta W_c}{2W_{max}} = \delta_s + \frac{3E_f h_f}{E_s h_s} \delta_f + \delta_{BG}. \tag{5}
\]

However, no systematic method has yet been proposed to quantify this BG. Moreover, some mechanisms of dissipation, including TED in the bilayer, cannot be easily partitioned by layer by layer [37]. To resolve all these long-standing problems, we propose a new approach based on an explicit mechanism-by-mechanism partitioning of \( \delta_c \). The essential idea underlying this approach is the use of TED for calibrating measurements of IF, as explained in Section II. The implementation of this approach using a single-crystal silicon microcantilever platform is described in Section III. Experimental results for IF in thin films of aluminum, gold, and silver are presented in Section IV.

II. THEORY: CALIBRATION USING TED

Consider a slender straight cantilevered beam of solid rectangular cross section with thickness \( h_s \) and length \( L_s \). The beam, which is free of residual stress, is micromachined such that it is attached monolithically to a larger supporting structure which, in turn, is clamped to the package or shaker. The beam and shaker assembly is located within a vacuum chamber that is maintained at a sufficiently low pressure to make viscous air damping negligible. The beam undergoes small-amplitude bending vibrations, and the structural damping can be measured from the free decay of bending vibrations and expressed in terms of the logarithmic decrement \( \delta_s \). Synthesizing results from several theoretical and experimental investigations of damping in vacuum-operated microcantilevers [1]–[3], [7]–[14], the measured damping can be partitioned into clamping losses, support losses, IF, and TED. Hence,

\[
\delta_c = \delta_{s,clamp} + \delta_{s,support} + \delta_{s,IF} + \delta_{s,TED}. \tag{6}
\]

The clamping losses are due to friction at the points of attachment of the vibrating structure to the test apparatus. This dissipation is difficult to quantify because it depends on numerous local variables at the region of contact. However, clamping losses can be minimized by following guidelines that have been developed based on experience. For instance,
involving a step at the base of the cantilever can minimize losses due to stick-slip friction [8]. The second source of dissipation is due to the interaction of the vibrating beam with the larger supporting structure. As the beam vibrates, it applies a time-harmonic force on the support, thereby leading to the generation and propagation of elastic stress waves into the support. Typically, the majority of this energy is not reflected back into the cantilever. Therefore, this mechanism of elastic stress wave generation is effectively a source of dissipation.

Analysis of an idealized geometry consisting of a cantilever attached monolithically to an elastic half-space predicts that stress wave generation is effectively a source of dissipation. Back into the cantilever. Therefore, this mechanism of elastic vibration is due to the coating of common metals (Al, Au, Ni, Cu, and Ag) on single-crystal silicon. In fact, the increase in TED due to the film cannot always be ignored even in the limit of a thin film on a thick substrate. As an example, the peak TED in a 100-µm-thick single-crystal silicon beam is $3 \times 10^{-4}$. If this beam is now coated with a 1-µm-thick film of nickel, the TED increases to $4 \times 10^{-4}$. This increase in damping is an order of magnitude larger than the resolution with which we seek to measure IF in the films. In fact, the increase in TED due to the coating of common metals (Al, Au, Ni, Cu, and Ag) on single-crystal silicon is negligible only if $h_f \leq 10^{-3} h_s$.

For such ultrathin films, $\delta_{c,TED} \simeq \delta_{s,TED}$; therefore, (9) can be expressed as

$$\delta_s = \delta_{s,TED} + O(10^{-5}). \quad (8)$$

Thus, silicon beams that follow (8) can be used to measure IF in deposited thin films with a resolution on the order of $10^{-5}$. To accomplish such measurements, we consider the deposition of a film of thickness $h_f$ on the silicon beam. If necessary, thin adhesion layers (Ti, Ta, and Cr with a thickness of $\sim 10$ nm) can be used to ensure good adhesion between the film and silicon [47]. The measured damping of this layered beam is the sum of TED in the composite and IF in the film. Hence,

$$\delta_c = \delta_{c,TED} + \frac{3E_f h_f}{E_s h_s} \delta_{f,IF} + O(10^{-5}). \quad (9)$$

The first term on the right-hand side captures the increase in TED due to the addition of the thin film. The frequency dependence of $\delta_{c,TED}$ can be computed accurately using a model developed by Prabhakar and Vengalator [48] that accounts for heat conduction across the thickness of the composite beam. This model leads to an expression for TED in the form of an infinite series. Although this expression is more tedious to evaluate than (7), it shares all the essential features of Zener’s formula, namely, there are no free parameters, and TED can be computed using only the material properties (Young’s modulus, coefficient of thermal expansion, specific heat, and thermal conductivity of the two materials) and the thickness of the two layers as input. Using this model, we found that the increase in TED due to the film cannot always be ignored even in the limit of a thin film on a thick substrate. As an example, the peak TED in a 100-µm-thick single-crystal silicon beam is $3 \times 10^{-4}$. If this beam is now coated with a 1-µm-thick film of nickel, the TED increases to $4 \times 10^{-4}$. This increase in damping is an order of magnitude larger than the resolution with which we seek to measure IF in the films. In fact, the increase in TED due to the coating of common metals (Al, Au, Ni, Cu, and Ag) on single-crystal silicon is negligible only if $h_f \leq 10^{-3} h_s$.

For such ultrathin films, $\delta_{c,TED} \simeq \delta_{s,TED}$; therefore, (9) can be expressed as

$$\delta_c = \delta_{s,TED} + \frac{3E_f h_f}{E_s h_s} \delta_{f,IF} + O(10^{-5}). \quad (10)$$

Equations (9) and (10) can be used to devise a new protocol to measure IF in thin films and ultrathin films, respectively. This protocol can be implemented using a sequence of several steps. The first step is to micromachine single-crystal silicon beams with carefully designed clamps and supports and to demonstrate that these beams can operate at the limit of dissipation established by TED. This is achieved by comparing the measured damping with the predictions of Zener’s formula (7).

The second step is to coat the beams with the films of interest. Independent measurements and guidelines based on experience are necessary to ensure excellent adhesion between the film microcantilevers with carefully designed supports and clamps. To the extent that IF, support losses, and clamping losses have been made negligible, we expect this class of beams to operate at the limit of dissipation established by TED. In practice, it is possible to reduce the combined contributions of IF, support losses, and clamping losses below $10^{-5}$ at room temperature [1]. Hence, (6) can be expressed as

$$\delta_s = \delta_{s,TED} + O(10^{-5}). \quad (8)$$
and substrate. The third step is to measure the damping in the bilayer. Finally, for ultrathin films of common metals, the IF in the film can be obtained from (10). For thicker films, the TED in the composite must be computed, and the IF in the film can be obtained by using (9).

This completes the development of the framework for measuring IF by using TED for calibration. The remaining sections of this paper describe the implementation of this approach to measure IF in thin films of aluminum, silver, and gold using a single-crystal silicon microcantilever platform.

III. MATERIALS AND METHODS

A. Micromachining Silicon Cantilevers

The starting materials for microfabrication were single-side-polished p-type (1 0 0)-oriented single-crystal silicon wafers with a thickness of \( \sim 520 \, \mu m \) and a diameter of 6 in. These wafers are doped with boron at a low concentration of \( \sim 10^{15} \, \text{cm}^{-3} \). First, the silicon wafers were oxidized for 26 min at 1100 °C to grow SiO\(_2\) films with a thickness of \( \sim 500 \, \text{nm} \) on both surfaces. The oxide films were then patterned using a standard photolithographic process and selectively etched; these patterns served as the mask for the anisotropic wet etching of silicon using tetramethylammonium hydroxide. The silicon wafer was diced parallel to [1 1 0]-direction and etched; these patterns served as the mask for the anisotropic wet etching of silicon using tetramethylammonium hydroxide. The etching of silicon wafers with a thickness of \( \sim 0.72 \) to 1.1 mm in width, and 0.21 to 3.6 cm in length. The length-to-thickness aspect ratio of the beams ranges from 58 to 834, and the fundamental frequency of bending vibrations ranges from 51 Hz to 10.9 kHz. A step was incorporated at the intersection of the beam with the support to reduce clamping losses (Fig. 1). Scanning electron microscopy (SEM) confirmed that the variation in thickness along the axis of the beams was less than 2%.

B. Sputter Deposition of Thin Metallic Films

Films of aluminum, gold, silver, chromium, and titanium were deposited using dc sputtering without actively heating or cooling the substrates and using metal targets with purity greater than 99.95%. In all cases, the thickness of the adhesion layers (Cr and Ti) was 15 nm. The thickness of the face-centered cubic (fcc) metals (Al, Au, and Ag) was varied between 50 and 500 nm to examine the effects of thickness on IF. The chamber was first pumped down to a pressure lower than \( 10^{-5} \, \text{torr} \) and then filled with argon to commence sputtering. Films were deposited on one surface of the single-crystal silicon microcantilevers. Care was taken to ensure that the clamping and supporting areas remained free of any coatings. This precaution is necessary to prevent any change in clamping losses due to metallization. In parallel, films were deposited under identical conditions on 4-in-diameter silicon wafers and used for characterizing the microstructure and residual stresses in the metallic films.

The thickness of the metallic films was measured using a surface profiler (Ambios XP 200, Ambios Technology, CA, USA) with a resolution of 1 nm. The deposited films were analyzed using SEM and tapping-mode atomic force microscopy (AFM) to assess the grain structure and surface topography. The intrinsic growth-related residual stress in the films was obtained by using Stoney’s formula to convert the measured change in wafer curvature (Flexus 5200, KLA-Tencor, CA, USA) [19]. Table I lists the deposition parameters, deposition rate, and representative values of the intrinsic stress. The stress in Al, Au, and Ag films ranges from 50 to 100 MPa in tension, and the stress in the 15-nm-thick adhesion layers was below the limit of resolution of the wafer curvature measurement.

In all cases, the metallic thin films were found to be polycrystalline with an average grain size on the order of film thickness. Thus, the average grain size ranged over one order of magnitude from 50 to 500 nm across the range of thickness examined in this paper. This variation of grain size with thickness is consistent with previous studies of microstructural evolution in sputtered thin films [47]. The root-mean-square roughness of the surface of the deposited films ranged between 5 and 20 nm, with roughness increasing with thickness, as inferred from the analysis of topographic images with an area of \( 1.5 \times 1.5 \, \mu m^2 \) obtained using AFM. A representative example of an AFM image is shown in Fig. 2.

C. Mechanical Spectroscopy Using Laser Doppler Vibrometry

The damping in the monolithic and composite cantilevered beams was measured as a function of frequency at room temperature by exciting the base of the beam using a piezoelectric shaker and measuring the dynamics of the cantilever using laser Doppler vibrometry. The cantilever beams were mounted on a comparatively large base clamp that was precision machined out of a single block of stainless steel. The flatness, parallelism, and the surface finish of the clamped surfaces were found to be important factors for minimizing clamping losses [1].

The clamp and cantilever assembly was then mounted onto a shaker consisting of a NanoOP65 piezoelectric positioner (NanoOP65, Mad City Labs Inc., WI, USA) that is driven by a function generator. A built-in electrostatic sensor permits accurate measurement and feedback control of the dynamics.
Table I

<table>
<thead>
<tr>
<th>Material (thickness)</th>
<th>DC Power Density (W/cm²)</th>
<th>Deposition Pressure (mtoorr)</th>
<th>Deposition Rate (nm/s)</th>
<th>Maximum stress (±15%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cr (15 nm)</td>
<td>2.4</td>
<td>25</td>
<td>3.0</td>
<td>&lt;40 MPa*</td>
</tr>
<tr>
<td>Ti (15 nm)</td>
<td>1.1</td>
<td>10</td>
<td>0.1</td>
<td>&lt;40 MPa*</td>
</tr>
<tr>
<td>Cr (15 nm) + Al (430 nm)</td>
<td>4.5</td>
<td>25</td>
<td>3.0</td>
<td>100 MPa</td>
</tr>
<tr>
<td>Ti (15 nm) + Ag (440 nm)</td>
<td>0.58</td>
<td>10</td>
<td>0.5</td>
<td>50 MPa</td>
</tr>
<tr>
<td>Ti (15 nm) + Au (440 nm)</td>
<td>0.58</td>
<td>10</td>
<td>0.5</td>
<td>75 MPa</td>
</tr>
</tbody>
</table>

*The stress in the thin adhesion layers was below the resolution of the measurement system.

Fig. 2. Topographic image of the surface of an aluminum film obtained using tapping-mode AFM. The film is 110 nm thick. Analysis of this 1.5 × 1.5 μm² area indicates a root-mean-square roughness of 8 nm.

Fig. 3. Photograph of the test apparatus used for measuring the dynamics of microcantilevers. (a) LDV, (b) vacuum chamber, and (c) shaker assembly with a carefully machined mount for the cantilevered specimen.

Fig. 4. Measurement of beam dynamics using laser Doppler vibrometry. The graph shows the tip velocity as a function of time during harmonic excitation and free decay. The inset shows the details of the velocity for a few cycles of vibration.
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Fig. 5. Frequency dependence of damping in 70 different single-crystal silicon microcantilevers at room temperature. The measurements are plotted as a function of the normalized frequency $\Omega$ defined in (7). Also shown for comparison is the curve predicted by Zener’s formula for TED.

Both methods lead to identical results for the logarithmic decrement of the beam. In addition, the slope of the decay envelope was examined to ensure that the damping was constant over the entire decay of the beam.

IV. RESULTS

Let us first examine the results obtained from measurements on single-crystal silicon microcantilevers. Seventy different silicon beams were micromachined and characterized. These microcantilevers range from 40 to 131 $\mu$m in thickness, 0.72 to 1.1 mm in width, and 0.21 to 3.6 cm in length. The length-to-thickness aspect ratio of the beams ranges from 58 to 834, and the fundamental frequency of bending vibrations ranges from 51 Hz to 10.9 kHz. The measured natural frequency for these beams was within 7% of estimates obtained from a standard analysis of the flexural vibrations of a slender elastic Euler–Bernoulli beam.

To assess the repeatability of the measurements, eight beams were selected and measured thrice. Each measurement included a complete remount, pump down, and three separate measurements of natural frequency and damping. Across these 72 measurements, the variations in the natural frequency and logarithmic decrement were less than 0.1% and 7.0%, respectively.

Fig. 5 shows a graph of the frequency dependence of the logarithmic decrement of the 70 different single-crystal silicon microcantilevers. These measurements are plotted as a function of the normalized frequency $\Omega$ defined in (7). Also shown on this graph is the curve corresponding to the prediction of Zener’s formula for TED in single-crystal silicon beams. This curve was computed by using (7) at 300 K with the following well-known values for single-crystal silicon: $E = 169$ GPa, $\alpha = 2.6 \times 10^{-6}$ K$^{-1}$, $C = 2.6 \times 10^6$ J/m$^3$/K, and $k = 149$ W/m/K [8]. The crystallographic orientation of the silicon beams was taken into account while selecting the value of the Young’s modulus [51]. In all cases, the measured damping approaches, but does not decrease below, TED; this behavior is consistent with the expectation that TED establishes the absolute lower bound on damping. The residual damping (that is, the difference between the measured damping and the predicted TED $\delta_s - \delta_{s, TED}$) ranges from $2 \times 10^{-6}$ to $3 \times 10^{-5}$, with an average value of $1 \times 10^{-5}$. This value establishes the resolution with which these silicon beams can be used to measure IF in deposited thin films.

Subsequently, a subset of 46 single-crystal silicon beams was selected and used to study the effects of metallization on damping. These beams range from 42 to 131 $\mu$m in thickness, 0.9 to 1.1 mm in width, and 0.9 to 3.6 cm in length. The length-to-thickness aspect ratio of these beams ranges from 88 to 557, and their fundamental natural frequency for bending vibration ranges from 97.5 to 1445 Hz. Six beams were used to quantify the effects of the 15-nm-thick adhesion layers of Cr and Ti on damping. Fig. 6 shows the measured values of damping in the bare silicon microcantilevers and in the Cr/Si and Ti/Si composites. The change in damping after metallization with the adhesion layers was below the resolution of measurement.

Next, we consider the effects of thickness and frequency on damping in thin films of aluminum, silver, and gold. In all cases, 15-nm-thick films of Cr and Ti were used to ensure adhesion of the fcc metals to silicon.

Fig. 7 shows the measured increase in damping $\delta_c - \delta_s$ as a function of the thickness of the fcc metals. All measurements were made at room temperature by exciting the composite microcantilevers at a frequency close to, but less than, their fundamental natural frequencies and then analyzing the free decay of these resonators. The fundamental natural frequency of the silicon beams that were used for these measurements ranged from 203 to 248 Hz. The change in frequency after coating with thin films (50 to 500 nm) of Al, Ag, and Au was measured to be negligibly small (typically, less than 0.5%). This is consistent with the predictions of a model for the vibrations of a slender bilayer consisting of a thin film on a thick substrate [21].
The results shown in Fig. 7 indicate that damping increases monotonically with film thickness. For any given thickness, aluminum dissipates more energy than gold. Indeed, the change in damping due to the 60-nm-thick gold film is at the limit of resolution of our technique. The same dependence on thickness was observed at all frequencies (97.5 to 1445 Hz) measured in this paper.

The change in damping after metallization was used to estimate the IF in the thin films. Using values from [37], we first checked that the change in TED due to metallization was negligible and then employed (10) to estimate IF in Al, Ag, and Au using the following values:

- $E_{\text{Al}} = 70$ GPa, $E_{\text{Au}} = 82$ GPa, $E_{\text{Ag}} = 76$ GPa, $E_{\text{Si}} = 169$ GPa,
- $\rho_{\text{Al}} = 2.7 \times 10^3$ kg/m$^3$, $\rho_{\text{Au}} = 1.93 \times 10^4$ kg/m$^3$, $\rho_{\text{Ag}} = 1.05 \times 10^4$ kg/m$^3$, and $\rho_{\text{Si}} = 2.3 \times 10^3$ kg/m$^3$.

Representative results for the effects of thickness and frequency on IF are shown in Figs. 8 and 9, respectively. IF in gold did not change significantly as a function of thickness. In contrast, IF in Al and Ag increased when the thickness was reduced below $\sim 200$ nm. Specifically, IF in silver doubled when the thickness was reduced from 230 to 60 nm. For both these metals, the highest value of IF was obtained at the lowest frequency ($\sim 100$ Hz) measured in this paper.

V. DISCUSSION

Let us first consider the results shown in Fig. 5 for single-crystal silicon microcantilevers. The comparison of the measured damping with predicted TED shows that silicon beams with carefully designed supports and clamps can approach the fundamental limits of dissipation set by TED. The residual damping $\delta_s - \delta_s,\ TED$ varies between $2 \times 10^{-6}$ and $3 \times 10^{-5}$ for the 70 beams shown in this graph. No correlation was found between this residual damping and beam geometry (length, thickness, and aspect ratios) or frequency, which suggests that support losses are not the main source for the residual damping.

Similarly, IF in silicon cannot be the source for the residual damping because all beams were micromachined using nominally identical single-crystal materials. By a process of elimination, we conclude that the residual damping is dominated by clamping losses. The average value of the residual damping is $1 \times 10^{-5}$, and this value sets the resolution with which this set of silicon beams can be used to measure IF in deposited thin films.

All silicon beams were coated with adhesion layers (15-nm-thick films of Cr or Ti) before metallization. Fig. 6 shows that the effects of these adhesion layers on damping are sufficiently small ($\sim 10^{-6}$) as to be negligible. In contrast, the damping in the metallized beams increased monotonically as the thickness of Al, Ag, and Au increased from 50 to 500 nm, as shown in Fig. 7. We conclude from this that dissipation is dominated by processes occurring within the film and not at the metal/silicon interface or at the free surface of the film. Indeed, if surface or interfacial processes were dominant, the damping in the
layered composite will be expected to be independent of film thickness. This was not observed at any frequency in our measurements.

The lack of dissipation due to interfacial sliding was expected because of the use of Ti and Cr to ensure excellent adhesion between the metals and silicon and also because of the lack of any significant shear stress at the metal/silicon interface. The latter is guaranteed by the geometry of a thin film on a thick substrate. In contrast, the absence of any significant surface dissipation in the metal films is unexpected and worth highlighting. Experiments on single-crystal silicon cantilevers, with thickness ranging from tens of nanometers to a few micrometers, have frequently identified surface dissipation as a major source of damping [2], [52], but the specific mechanisms responsible for energy loss at the surface have not yet been identified.

To our knowledge, the results presented in this paper are the first measurements of the effects of thickness and frequency on IF at room temperature in thin films of aluminum, silver, and gold. These results provide some useful guidelines for designing layered resonators for MEMS: Gold leads to a smaller increase in damping than either aluminum or silver, and damping in the composite resonators can be decreased by reducing the film thickness. These conclusions are based on measurements at relatively low frequencies (100 Hz to 1.5 kHz), and additional studies are necessary to confirm whether the same trends hold at higher frequencies.

It is difficult to make a detailed quantitative comparison of our results on IF with those reported earlier in the literature because of crucial differences in technique, test conditions, and geometry. Previous studies have focused mostly on exploring the effects of temperature on IF at a few values of thickness and frequency. Moreover, these studies have focused typically on films with thickness on the order of 1 μm in contrast to our measurements on thinner films (50 to 500 nm). All our measurements are from beams vibrating in the fundamental mode; we have not accessed higher frequencies by exciting higher modes of vibration, as is often the case in many other studies. Finally, our technique relies upon the use of TED for calibration and an explicit mechanism-by-mechanism partitioning of the dissipated energy. In contrast, all previous studies have used a layer-by-layer partitioning following (4) or (5) and have not accounted for TED in the composite.

Nevertheless, we can attempt a qualitative comparison with some earlier studies. Liu et al. [53] measured damping in thin films deposited on silicon by using double-paddle torsional oscillators vibrating at 5.5 kHz. The IF in evaporated films of gold with thicknesses of 10, 31, 106, and 310 nm did not change significantly with thickness at temperatures ranging from 0.5 K to 100 K. The second comparison is with a set of studies of IF in aluminum films at high temperatures [23], [28], [34]. The IF consists of a temperature-dependent background (presumably due to dislocation activity) and a peak attributed to grain-boundary sliding. It is therefore possible that both these mechanisms contribute to the IF measured in the thin Al films in this paper. However, the relative dominance of these two mechanisms at room temperature is not currently known.

VI. SUMMARY

This paper has presented a new approach for accurate measurement of IF in deposited thin films. At the heart of this approach reside the ability to operate single-crystal silicon cantilevers at the fundamental limit of dissipation established by TED and the ability to compute TED in film-coated silicon cantilever resonators using only material properties and geometry as input. Using this approach, we measured IF at room temperature in sputtered polycrystalline films of aluminum, gold, and silver with thickness ranging from 50 to 500 nm at frequencies ranging from 100 Hz to 1 kHz. In all cases, IF is due to processes occurring within the film and not at the film/silicon interface or the free surface of the metal. The IF in aluminum is consistently higher than that exhibited by gold.

This technique can be used to develop accurate database of IF in thin films at room temperature to guide the selection of materials for micro/nanomechanical resonators and to inspire models for the mechanisms of defect-induced dissipation. Extending the technique to higher frequencies in the megahertz range and to higher temperatures of a few hundred degrees Celsius is a major focus of our current efforts.

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REFERENCES


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