Anelastic Mechanical Behavior Measurement of thin Films Using Novel Beam deflection Method

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Abstract

In this paper a technique developed for studying the anelastic behavior of nano-scale thin metal films on substrate is presented. The test microstructure was designed the triangular cantilever beam and fabricated by the standard C-MOS processes, which can improve stress distribution non-uniform problem and the thickness regime of deposited metal thin film on its surface could reduce to several nanometers. In order to reduce the measure error and calculation complex due to the contact force (like indenter), the driving system was used electrostatic force to making the paddle cantilever beam bend and the deflection of paddle cantilever beam due to the electrostatic force was measured by a capacitance change (Fig. 1). The deflection of the paddle beam can be measured from the capacitance value. A force equilibrium calculate method (include sample compliance force, force due to the film, force due to the gravity and electrostatic force) could determine the stress and strain of the deposited films easily. The anelastic behavior and internal friction study of 200~500 nm Ag & Al thin film were studied using the dynamic frequency response of the paddle structure generated by electrostatic force under vacuum pressure. The results in loss mechanism show evidences of grain boundary motion and dislocation motion in the film.

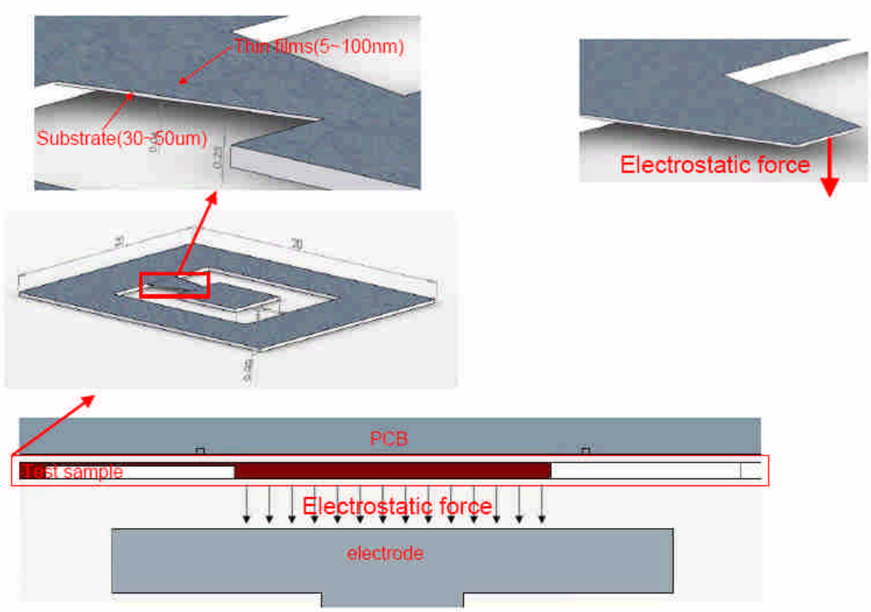

Fig. 1. Schematic of Measurement System

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Abstract. In this paper a technique developed for studying the anelastic behavior of nano-scale thin metal films on substrate is presented. The test microstructure was designed the triangular cantilever beam and fabricated by the standard C-MOS processes, which can improve stress distribution non-uniform problem and the thickness regime of deposited metal thin film on its surface could reduce to several nanometers. In order to reduce the measure error and calculation complex due to the contact force (like indenter), the driving system was used electrostatic force to making the paddle cantilever beam bend and the deflection of paddle cantilever beam due to the electrostatic force was measured by a capacitance change (Fig. 1). The deflection of the paddle beam can be measured from the capacitance value. A force equilibrium calculate method (include sample compliance force, force due to the film, force due to the gravity and electrostatic force) could determine the stress and strain of the deposited films easily. The anelastic behavior and internal friction of 200~500 nm Al thin film were studied using the dynamic frequency response of the paddle structure generated by electrostatic force under vacuum pressure. The result show the measurement system used here can accurately measures the loss mechanism of thin film using dynamic response which give potential to study the grain boundary motion and dislocation motion in the nano-scale thin films.

1 Introduction

In recent years, with the vast development of the micro-electro-mechanical systems (MEMS) technology, the further miniaturization of thin films is required in order to increase the performance need and cost efficiency. As a result, the mechanical properties of sub micron and nano scale thin films have become one of the most important issues. In MEMS applications, the mechanical properties such as residual stress, modulus and fractural toughness are keys for the design protocol. Moreover, anelastic behavior of metal thin films as a function of operation time can be pivotal. However, due to the difficulties on measurement techniques, a simple and accurate measurement arrangement cannot be fulfill [1].

Many methods have been proposed for measuring the mechanical properties of thin film materials. These methods can divides into two parts. The first part is the thin film attached to a substrate, such as wafer curvature, nano-indentation and lattice strain measurements. The second approach measures suspended thin film itself after removing it from the substrate; such as the bulge test, micro-beam bending test, micro-tensile test and resonance methods. All of these methods can be used to measure the mechanical properties. However, there are issues that need to be overcome with

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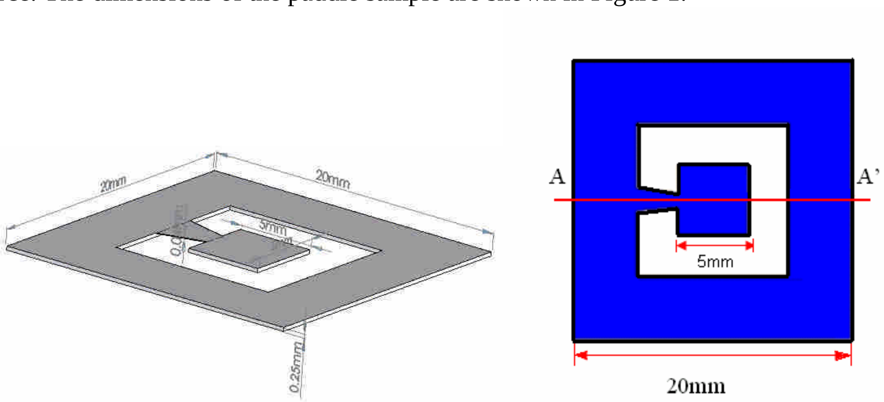
each method. For example, in the traditional micro-beam bending test, the applied force and deflection are measured using nano-indentation. A cantilever beam with a single point load at the free end is used to measure the stress-strain relation from the relation between the deflection and the load. The cantilever beam with a parallel side sample would cause the stress distribution to be non-uniform. In addition, the indenter tip touching the sample surface may break the thin film. Therefore, the results obtained from different measurement techniques were vary widely for nominally identical samples due to the difficulty with the techniques [2, 3].

In the literature reviews on dynamic damping responds of materials, many anelastic mechanisms had investigated in bulk material [3], but rarely in thin films. In order to understanding the accurate dynamic response of the thin film materials, the energy dissipation study through simply damping response of thin film on substrate was performed. This paper developed a method that can be used to measure the anelastic dynamic behaviors of thin metal films with thickness less than 100 nm. The test specimen was designed to deposit on a novel triangle shape "paddle" beam in order to provide uniform plane strain distribution. When the sample reached the desired thickness, the tested thin film on the top surface can then be tested for measuring its static and dynamic mechanical properties.

2 Sample design and fabrication

A novel paddle structure was presented in this paper. A triangular shaped beam was designed to provide a constant surface stress using electrostatic force to avoid the measurement error due to contact force. The dimensions of the paddle sample are shown in Figure 1.



(a) **(b)**

Fig. 1. The dimensions of the sample (a) overview (b) top view.

The sample dimensions are 20mm × 20mm, using a silicon substrate. The length of the triangular beam from the fixed end to the free end connected to the paddle plate is 3mm. The area of the paddle plate is 25mm^2. The thickness of the silicon wafer is 250μm. The thickness of the cantilever beam is 40μm after etching. All of the test structures were fabricated using standard semiconductor processing. Double sided polished silicon 4' wafer through the RCA clean process for removing particles and organisms. Silicon nitride about 200~300 nm was grown on both surfaces of the wafer using low pressure chemical vapor deposition (LPCVD). Photolithography is used to pattern the photoresist layer onto both sides of the non-protect zone. Silicon nitride was etched using ICP-RIE and the photoresist removed using acetone. The silicon wafer was etched until the beam thickness was 40um using a hot KOH solution. When the paddle beam reached the desired thickness, the silicon nitride layer was removed using HF solution. Sputter deposition was used to deposit the metal conduct layer onto the bottom surface. The thin film on the top surface was then measured.

The complete fabrication process flow is shown in Figure 2. A photo of the sample after the complete process is shown in Figure 3.

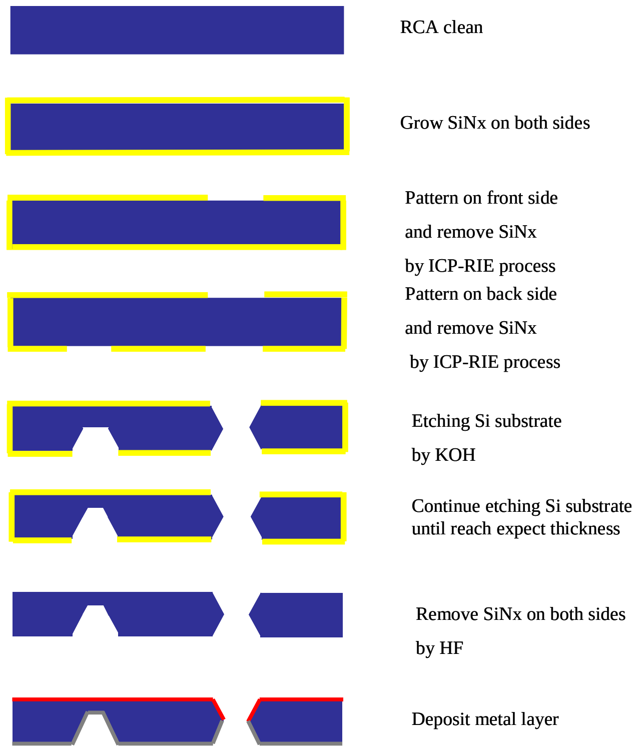


Fig. 2. The fabrication processes of the sample.

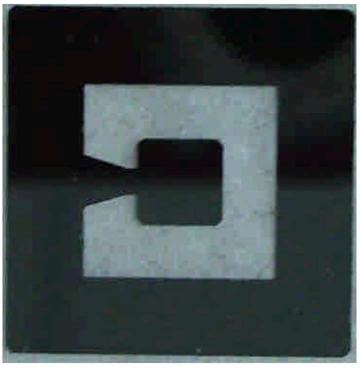


Fig. 3. The schematic of the paddle sample.

3 Measurement systems

The experiment is to deflect "paddle" cantilever beam sample using the electrostatic force at bottom and then using capacitance measurement on the other side of the deflected plate to measure its deflection with respect to the force. This technique has allowed testing of thin films at the desired length scales with the thickness as few hundred nanometers to less than 10 nanometers and also maintained consistent preparation and experimental procedures.

The system design is shown in the schematic of Figure 4. It consists of the guard-ringed capacitor electrode, the metal spacer, the calibration sample and the deflection electrode. During the experiment, we apply an electrostatic force to the specimen and measure the capacitance change. The deflection of the paddle beam can be measured from the capacitance value. The sample chip is mounted together with a guard-ringed capacitor electrode as shown in Figure 5. A spacing of 25 to 125um to the window frame chip surface around the paddle structure is defined by a metallic spacer.

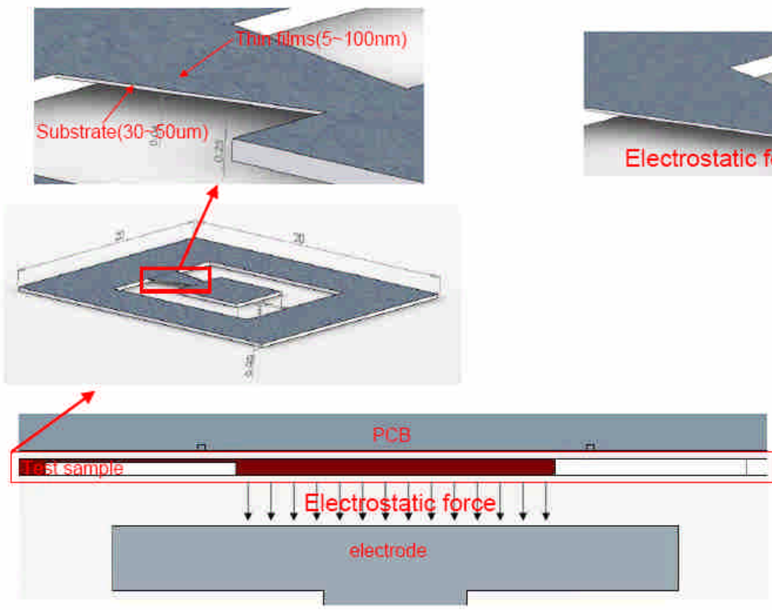


Fig. 4. The schematic of the system design.

A second electrode is mounted below the paddle plate. This electrode is used for electrostatic deflection of paddle. The whole chip is at DC ground but driven at 100 kHz with amplitude of a few volts. That provides a displacement current to central electrode of the capacitor plate which is proportional to the capacitance, and hence inversely proportional to the gap. Depending on the spacing selected, the capacitance is between 2 and 4 pF. The measurement of the capacitance can be made to a precision of approximately 0.1 fF so that paddle spacing changes of 50 nm are readily determined. The paddle can be pulled up with a DC voltage on the guard-ringed electrode or pulled down with a DC voltage on the lower electrode. The capacitance measurement can be made with a time resolution of ±10 msec.

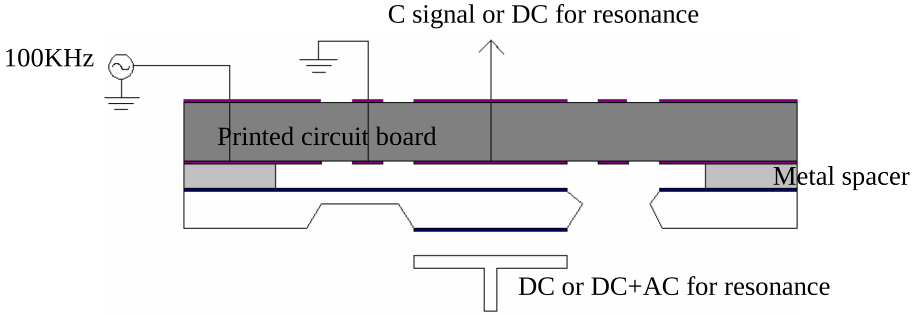


Fig. 5. The schematic of the measurement design.

For the electronic setup of the capacity measurement, a sine-wave generator at 100 kHz is applied to the film simultaneously measuring capacity of the paddle capacitor while a second generator drives at the same frequency for a test capacitor which has a known capacity. The two units are coupled (one master, one slave) and have a phase shift of 180^0. Figure 6 shows the circuits. The 180^0 out of phase currents from the two generator-capacity pairs are summed at input of change sensitive preamplifier. The amplified sum is measured with lock-in amplifier with the reference signal from one of the frequency generators.

When the current flowing through the two capacitors is approximately equal, the lock-in will show a value close to zero. In this case the ratio of the capacities is inversely-proportional to the ratio of the amplitudes of the driving:

$$V_1 \cdot C_1 \sim \frac{V_1}{Z_1} = \frac{V_2}{Z_2} \sim V_2 \cdot C_2 \qquad (1)$$

If now the capacitance of the paddle capacitor is changed by a change in y_p, the 180^0 out of phase currents are unbalanced and the lock-in amplifier will measure the difference.

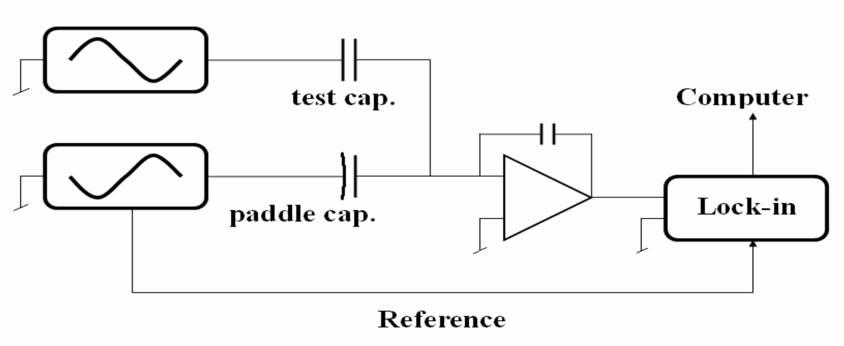


Fig. 6. The electronic setup for the capacity measurement.

The electrical driving circuits system was based on the charging parallel-plate principle when the two parallel planes shift produces the electrical field. Here, the two charging plates in the system were both the bottom surface of the sample and the electrode underneath the paddle plate. It is defined the electrostatic force

$$F_e = \frac{1}{2}\varepsilon_0 \frac{V^2}{d^2} A \qquad (2)$$

where, F_e is the electrostatic force, ε_0 is the dielectric constant in the vacuum, V is the applied voltage, d is the distance between the two parallel plates and A is the effective area of the parallel plates.

When the paddle beam is bending, the distance between the two plates is not the constant but is a function of x. Therefore, equation (2) is rewritten as equation (3).

$$F_e = \frac{\varepsilon_0 l_p}{2} \int_0^{l_p} \frac{V^2 dx}{(d_e + y_b + slope \cdot x)^2} \qquad (3)$$

where F_e is the electrostatic force, ε_0 is the dielectric constant in the vacuum, l_b is the length of the paddle cantilever beam, l_p is the length of the paddle plate, V is the applied voltage, d_e is the distance between the under surface of the sample and the electrode, and y_b is the position of the paddle cantilever end. The symbol in the cantilever beam and the test sample structure are shown in the Figure 7.

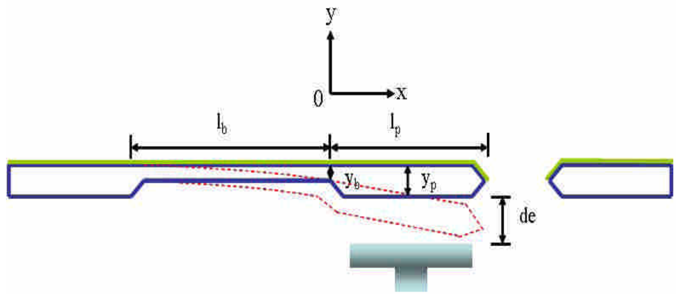


Fig. 7. The symbol definition of measure system.

Integrating equation (3) and rewriting it as equation (4), the electrostatic force is defined as

$$\frac{F_e}{V^2} = \frac{\varepsilon_0 l_p l_b}{4 y_b} \left(\frac{1}{d_e + y_b} - \frac{1}{d_e + y_b + \dfrac{2 y_b l_p}{l_b}} \right) \qquad (4)$$

The measurement is controlled by PC through National Instrument LabVIEW program. The control electronics include a controller, amplifier and waveform generator. Monitored signals are conditioned and then fed into an A/D board which is located in a PC. Data acquisition is performed with LabVIEW software. During sample testing, it is placed inside the chamber, and the samples are monitored with an optical microscope. After the sample is being locked inside the system, then wait until the system is reaching the thermal equilibrium and the capacitor read out is clear, the test can be perform.

Before the measurement, a position calibration sample with the beam thickness the same as the rectangle plane at full wafer thickness was produced. This sample was used to confirm the original position of the paddle beam without bend. The non-coated sample was then mounted onto the steady system and driven to 125 volts to measure the deflection in situ. The data was plotted as the blue line in Figure 8. After this step, the sample was deposited with aluminum thin film on its top surface and voltage applied from 0 to 125 volts. The applied voltage and the sample deflection were recorded using DAQ hardware and plotted as the red line in Figure 8. Figure 8 compares the non-coated sample with the coated sample to determine the residual stress. The aluminum film is tensile stress, because the position of y_p with the coating film sample is higher than without the coating.

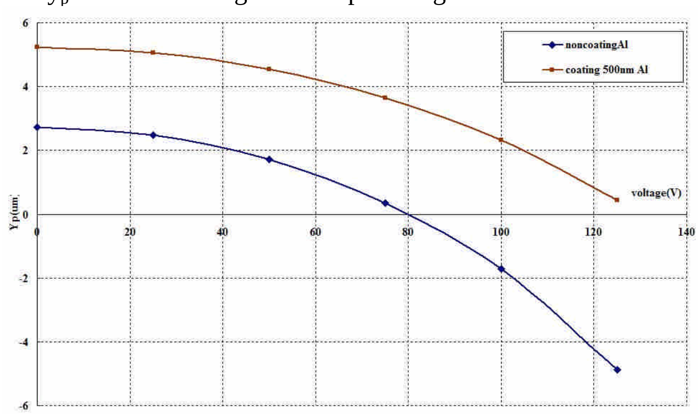


Fig. 8. The comparison of Yp versus applied voltage for sample with and without thin film

Figure 8 showed the measurement of the sample with and without coating the aluminum thin film. The thickness of the constant stress cantilever beam is thinner than the rectangle paddle plane, so the weight of the paddle plate may cause constant stress in the beam producing a little bending due to gravity. The spring constant of the samples were calibrated before tested. A known weight was given under the center of paddle plate and then deflection was measured with respect to the known weight. This procedure was performed both on loading and unloading to check the consistency. Here, the spring constant of the 40μm paddle cantilever beam is 0.0339mN/μm.

The study utilizes displacement height measurement based on the electrostatic deflection of “paddle” cantilever beam has allowed testing of the thin films at the desired length scales and also maintained consistent preparation and experimental procedures.

4 Results and discussions

For the anelastic and viscoelastic measurement of thin film, it is to observe the stress relaxation behavior of tested thin films at any given constant temperature through both static and dynamic measurements. In each constant temperature, we first measure the capacitance of the sample with no voltage on the deflection plate, and then apply a voltage to the deflection plate either suddenly or as a rapid voltage ramp to a final value and hold at that value. This adds an incremental tensile strain to the film. Then we can measure the capacitance as a function of time. As the film creeps, the film stress will decrease and the paddle will deflect and reducing the capacitance. After a time, return the deflection voltage to zero. Again, we measure the capacitance as a function of time as a result of the incremental compressive strain that is created. The equivalence of the transients after the two incremental strains will indicate viscoelasticity of the film. By carrying out the sequence of measurements at a variety of temperatures respect to its activation energy, the relaxation process of the sample should be obtained. With appropriate feedback between the capacitance measurement and the applied voltage, we can possibly maintain a constant strain and measure the stress relaxation as revealed by the electrostatic force required in the feedback. The experiment could be carried out with different film thicknesses to look for thickness effects, with different interfaces or with alloying constituents added to the metal of the film. Moreover, given the fact that any grain boundary motion and dislocation motion in the film will extract the energy, we can observed those behavior through the internal friction measurement of the sample both in room temperature.

The anelastic mechanical behavior measurement of thin films dynamic properties of thin film were studied using the dynamic frequency response of the paddle structure generated by electrostatic force. It was using an applied step voltage on the deflection electrode and then the elastic aftereffect behavior was observed. We observed the paddle sample returns to its initial position with free damping when the electrostatic force was suddenly removed from the paddle sample and the amplitude of the damping response was decay with time. Figure 9 shows the results of the damping behavior for the paddle sample with 300nm aluminum film tested in the vacuum (1.6E-2 torr).

Internal friction, δ, is defined as the ratio of the energy dissipated per cycle and the maximum elastic energy stored in one cycle. The dissipation of elastic energy in a vibrating sample caused by stress induced defect motion is rate-limited by a kinetic process. Usually, internal friction of vibrating samples can be determined from the rate of decay of the amplitude. From the frequency and free decay data, we can calculate the internal friction from a simply equation:

$$y(t) = y_0 e^{-n} = y_0 e^{-\delta \cdot fot} \qquad (5)$$

where y(t) is the vibration amplitude, yo is the initial amplitude, n is the decrement of damping, δ is the internal friction parameter, fo is the damping frequency. Table 1 summarized of internal friction parameters measured in this system with different film thicknesses. Table 2 summarized of the decrement of damping parameters measured in this system with different vacuum pressure.

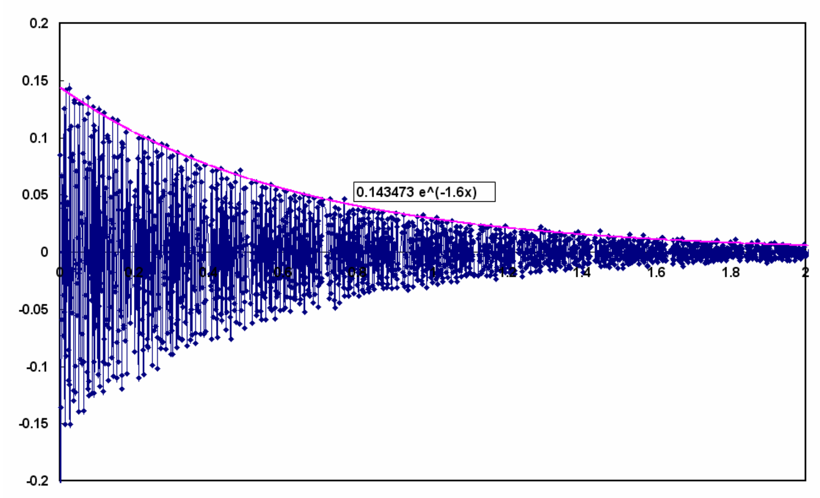


Fig. 9. Free decay of sample after one step voltage was applied on bottom electrode and recorded from the lockin output.

Table 1. Summary of internal friction parameters measured in this system with different film thicknesses.

	Nocoating	100nm Al film	300nm Al film	500nm Al film	1000nm Al film
fo(Hz) in the air	229.78	216.56	224.29	181.81	229.78
fo(Hz) in the vacuum	234.78	222.31	234.34	181.66	231.66
n in the air	154.98	184.21	137.47	101.02	91.249
n in the vacuum	1.5118	0.3604	0.3277	0.3411	0.2770
δ in the air	0.6745	0.8506	0.6129	0.5556	0.3971
δ in the vacuum	0.0064	0.0016	0.0014	0.0019	0.0012

Table 2. Summary of the decrement of damping parameters measured in this system with different vacuum pressure.

Pressure (torr)	Film thickness(nm)	Fo	n	δ
$1.6\text{x}10^{-2}$	100	222.31	0.3604	0.0012
$2.1\text{x}10^{-2}$	100	222.53	0.5765	0.0026
$5\text{x}10^{-2}$	100	224.3	1.1126	0.0050
$1.6\text{x}10^{-2}$	500	181.66	0.3411	0.0019
$1.7\text{x}10^{-2}$	500	181.66	0.3659	0.0020
$1.8\text{x}10^{-2}$	500	181.66	0.3702	0.0020
$1.9\text{x}10^{-2}$	500	183.17	0.4015	0.0022

The damping time constant has significant different between test in air and vacuum. It is clear the air damping reduces damping factor Q in the order of 3. Figure 10 shows the decay rate after suddenly removing the applied voltage of the sample at different pressure, the damping time is less than 0.06 seconds for the paddle sample tested in the air and the free damping decay time took more while vibrated in the low vacuum environment.

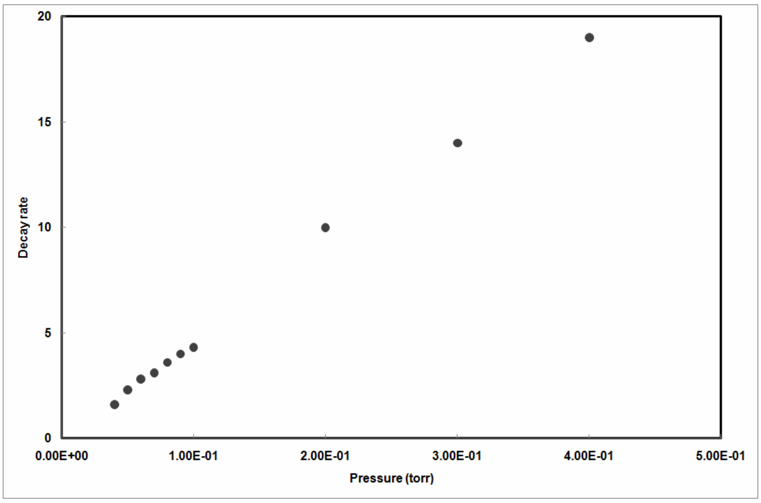


Fig. 10. Free decay rate versus operation pressure.

Figure 11 shows the frequency scan of the tested paddle sample with 500nm Al film deposited on top. This result shows the shift comparison of resonance frequency triggered by the electrostatic force. Fig. 12 shows the frequency scan of the tested paddle sample with four different thickness of Al film deposited on top. Fig. 13 shows the frequency scan of the tested paddle sample with four different thickness of Al film deposited on top. Fig. 14 shows the fast Fourier transforms plot for the analysis of the power frequency spectrum. These results show the measurement system used here can accurately measures the loss mechanism of thin film using dynamic response which give clear observation for the study of the grain boundary motion and dislocation motion in the film.

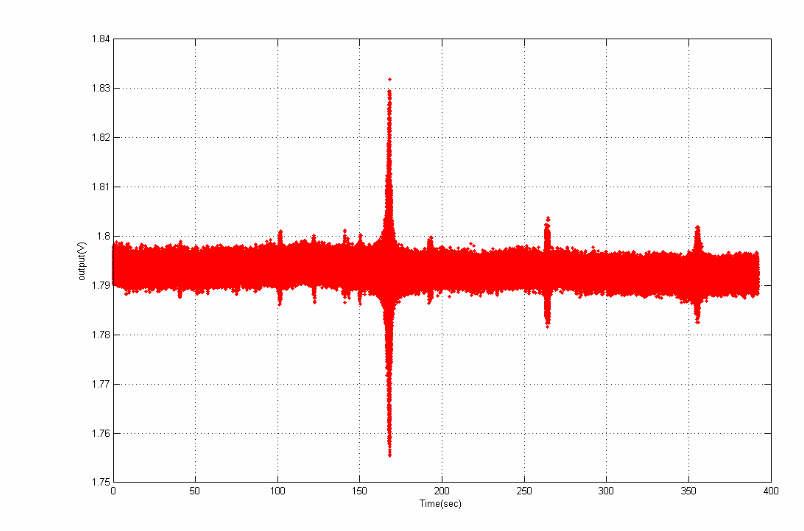


Fig. 11. The frequency scan for the tested paddle sample with 500nm Al film deposited on top.

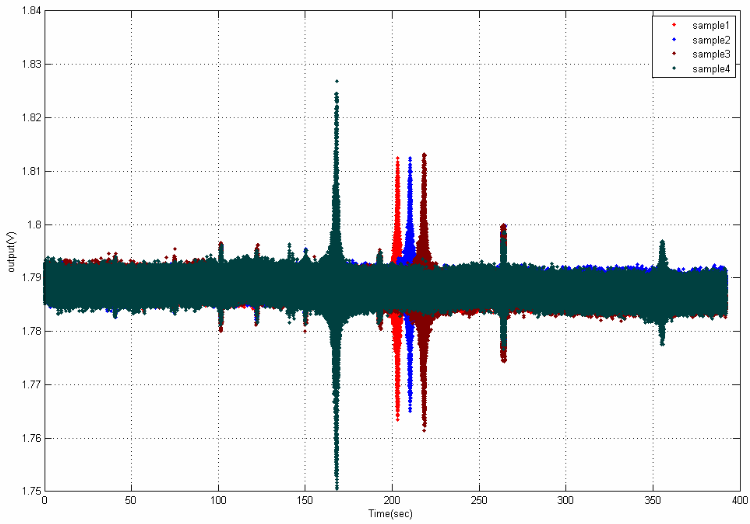


Fig. 12. The resonant frequencies scan using sweep frequency for four different samples.

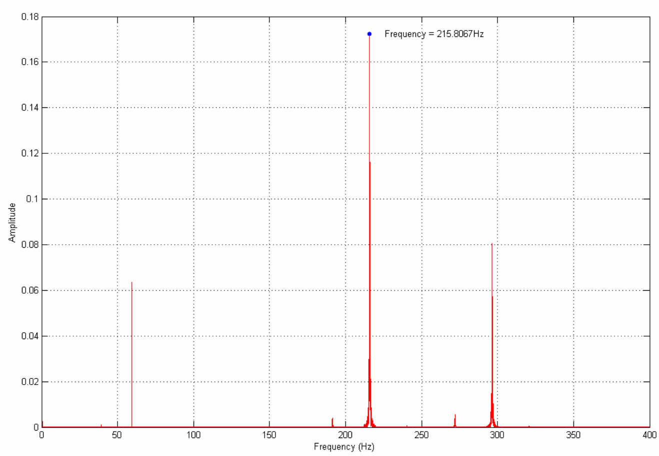


Fig. 13. The fast Fourier transforms plot for the analysis of the power frequency spectrum.

5 Conclusions

A novel paddle cantilever beam was successfully calibrated and fabricated. The electrostatic force caused the beam to bend and the deflection of various aluminum films was measured. The deflection versus the electrostatic force translated the stress and strain relation and dynamic damping of the thin film was observed. These results indicate that the decrement of damping is increased with the pressure increased and the air damping effect is significant in damping response. In addition, the film thickness effects on resonance frequency of vibrated sample were observed. These results show the measurement system used here can accurately measures the loss mechanism of thin film using dynamic response which give potential to study the grain boundary motion and dislocation motion in the nano-scale thin films.

Acknowledgment

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