Vibrating membrane elastometer for reliable measurement of mechanical properties of metallic films

A. Fartash
Physics Department, B-019, University of California, San Diego, La Jolla, California 92093 and Materials Science Division, Argonne National Laboratory, Argonne, Illinois 60439

Ivan K. Schuller
Physics Department, B-019, University of California, San Diego, La Jolla, California 92093

M. Grimsditch
Materials Science Division, Argonne National Laboratory, Argonne, Illinois 60439

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We describe an apparatus for measuring the biaxial mechanical properties of metallic films. A circular knife-edge support forms a drum-head geometry over which the film to be studied is stretched. The film's tension is obtained from the frequency of its membrane modes, and its strain by using an optical technique. The biaxial modulus is obtained from the ratio of tension and strain. An electronic detection system is developed that regeneratively locks to the film's resonant frequency. The system is also capable of measuring the film's damping when the lock is switched off.

I. INTRODUCTION

The mechanical properties of metallic films have been of growing interest in the past few years.[1-8] A number of mechanical techniques have been developed to measure the elastic moduli and damping of metallic films. Because thin films are easily deformed experimental artifacts arise that make most techniques unreliable.[8-10] For instance, "vibrating reed" can produce results that for a given material can vary by factors as large as 2–3.[3] "Bulge tester" measures the biaxial modulus of thin films but is sensitive to their initial configuration and warpings; as a consequence non-Hookian stress-strain behavior may result if initial deformations are not properly accounted for.[1,9] "Nano-indenter" is a device that measures both the elastic and plastic properties of thin films. However, local plastic deformations caused by the indenting stylus, and the influence of the substrate make the interpretation of results difficult.[10] In this article we describe a novel apparatus designed to produce reliable and reproducible results. The reliability of our technique has been briefly discussed in Ref. 7; here we present a complete description of our equipment and also additional experimental results. The apparatus consists of two main parts; a mechanical assembly that tensions the film as it stretches it over a circular knife-edge support, and allows optical determination of the strain. Second, an electronic system that provides rapid measurement of tension and damping of the film.

II. MECHANICAL ASSEMBLY

A. Description

The proper handling of a self-supporting film requires distinction of three important steps: (i) rigid clamping, to hold the film firmly in place to avoid lateral movements during its measurements, (ii) light stretching, to clear initial warpings and wrinkles so that the film is configured as a membrane modeled in the analysis, (iii) uniform tensioning, to vary the biaxial strain in the film. Figure 1 shows the cross-sectional view of the drum head assembly used in this study which allows the three steps mentioned above to be performed.

The drum-head assembly is machined from brass. It seals through a rubber O-ring "O" to hold vacuum against an aluminum chamber "V" (not shown). The chamber is evacuated to reduce the damping caused by air on vibrations of the film.[11] For this purpose a sorption pump is used to minimize low frequency disturbances. To accomplish step (i) mentioned above the metal film ("F" in Fig. 1) is clamped between two brass pieces "B" and "C"; these pieces have surfaces with a coarse finish to enhance their grip on the film. Steps (ii) and (iii) are achieved by the piece "A" which is also machined from brass and has a circular finger that presses against the film. This finger stretches the film over 3/8-in.-diam circular knife-edge support labeled "C." The force exerted by "A" is controlled by three stainless rods "SR." These rods are vacuum sealed by teflon feedthroughs "T," and attached to a control plate "CP" whose movement is in turn controlled by a micrometer "M" with its tip pressed against a stainless-steel ball "SB." The strain in the film is varied by turning the micrometer and measured optically by viewing the film through a high magnification microscope. The objective of the microscope is positioned beneath the assembly facing a glass window "GW" (not shown) sealed with rubber cement to "V." The assembly is free to move in three perpendicular directions controlled by three independent micrometers (see Fig. 2). A rectangular glass reticule "R" which faces the film enables the viewer to track specific markings on the film and thus measure induced displacement to determine the strain. To reduce stray reflections "GW" and "R" both have multilayer antireflection coatings and "A" is sand blasted and coated with a black enamel (on its side facing the microscope's objective).

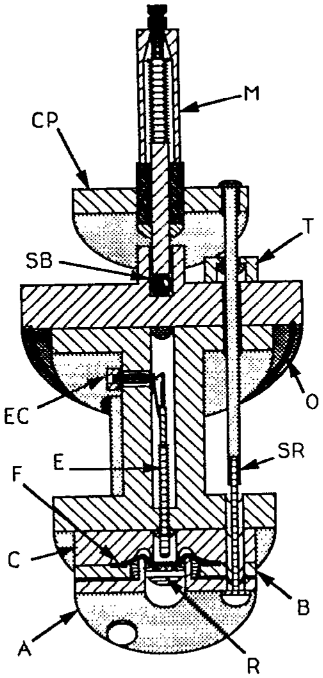

FIG. 1. Cutaway schematic for the sample-holder assembly. A small circular nickel plate (1/8 in diameter) is attached to the center of the reticule "R" (not shown).

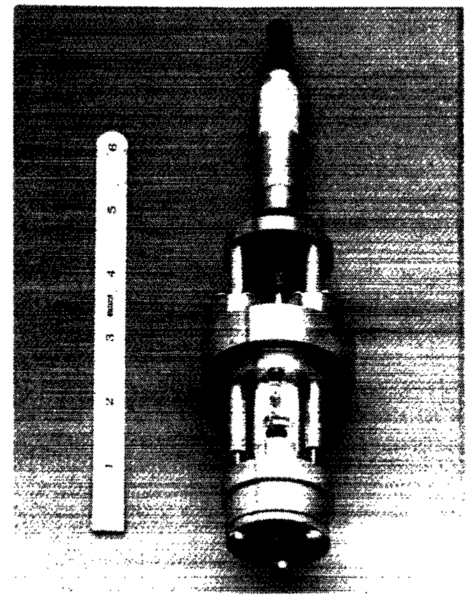

FIG. 3. Sample holder assembly. With respect to the schematic in Fig. 1 it has been rotated 90° around its long axis.

Since the microscope is operated in reflection mode, the sample is thus illuminated through the microscope's objective.

The applied tension is determined from the fundamental mode of the vibrating drum-head membrane. The vibrations are induced electrostatically by a thin nickel plate ($\sim 1/8$-in. diameter, $\sim 1/32$ in. below the vibrating film) glued to the center of the glass reticule "R" facing the film. A copper wire is attached to the nickel plate to which an oscillating voltage is applied. An ac coil can also be used to drive the film if a large driving force is desired.[12] The resonances are detected by an electrode "E" facing the film on its other side. The tip of "E" forms a capacitor with the film with a spacing of ~ 0.5 mm. "E" has a guarded shield to reduce its stray capacitance and is isolated from the ground assembly by telfon spacers.

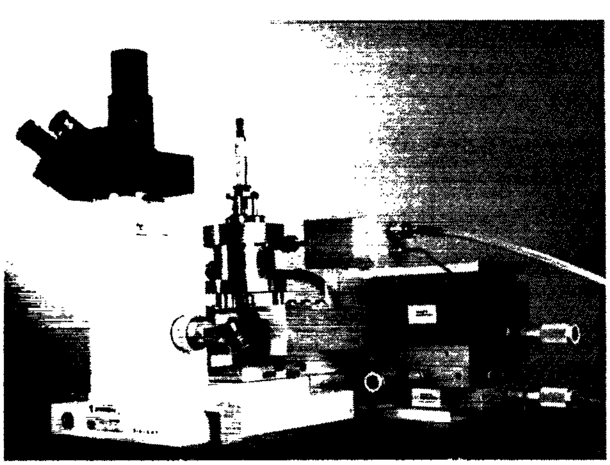

FIG. 2. Experimental setup consisting of an evacuated cylindrical chamber positioned on a platform above the microscope objective. The pre-amp (with its output cable) is directly attached to the receiver electrode connecter, EC.

Figure 2 shows the experimental setup. The sample holder (Figs. 1 and 2) is positioned in the vacuum chamber "V" that rests on an aluminum platform above the microscope's objective. Figure 3 shows the picture of the sample holder alone.

B. Operation

A metal film is first cut into a $\sim 3/4$-in.-diam piece and carefully deburred. It is subsequently placed between the drum-head pieces "B" and "C." Six brass screws are tightened to clamp the film rigidly in place. "A" is then gently positioned on the film to apply slight tension to make it flat. Three aluminum spacers are inserted between "A" and "B" to hold them uniformly apart and the control rods "SR" are locked, to snugly hold "A" in place. At this point the spacers are carefully removed and the sample holder is put into the chamber "V" for inspection through the microscope. After initial checks, the electrode connector "EC" is inserted and the chamber is pumped out. On the left and right ends of the film specific markings are chosen and their positions on the microscope reticule are recorded with respect to the markings on the glass reticule "R." The tension is then increased until the impression of the knife boundary is visible on the film. After this procedure the resonant frequency and induced displacements are measured each time the tension is varied. With increasing tension the resonant frequencies increase; when the tension reaches the film's tensile strength a decline in frequency signifies the break up of the film. The procedure is repeated with a new film, but this time the tension is kept below the tensile strength. To avoid problems resulting from plastic deformation the film's elastic response is measured as tension is released. The measured strain versus tension represents the elastic behavior of the film; the slope of a least squares fitted line gives the biaxial modulus.

The frequency of the fundamental mode (ν_0) versus tension for a circular membrane of radius a is given by[7]

$$T=\rho(2.613a\nu_0)^2, \tag{1}$$

where ρ is the density. The equation is valid in the limit when the restoring force is dominated by tension, T (force per unit area), at the film's boundary and the plate rigidity has a negligible effect. Since in these experiments tension is released from large values (close to the film's tensile strength), for most films the tension is sufficiently large for the film to behave as a membrane.

To verify if the film behaves like a membrane, an internal consistency check is always carried out. For this purpose the excitation frequency is swept through a number higher modes and the resonances are compared with theory. If membrane properties dominate, the higher modes (the first three) are within a few percent from the calculated values. As multiples of ν_0 the modes are given as follows: 1.5933, 2.1355, 2.2954, etc. If the film is too thick (or tension is too small) so that it behaves like a plate, the ratios to the first mode are 2.091, 3.426, 3.909, etc.[7]

The above internal consistency check also yields information on the uniformity of the tension at the film's boundary. An excessive splitting of a degenerate mode (the second and third modes are doubly degenerate) signifies uneven tension,. Usually large splittings are caused by initial warpings of the film. A careful examination of the mechanical design reveals that nonuniform tensions are not due to canting of piece "A" but to either wrinkles in the initial mounting or anisotropic internal tensions in the film.

III. ELECTRONIC SYSTEM

A. Description

The electronic system is composed of modular units each of which performs an independent function. The main function of the system is to drive and detect the film vibrations. In addition, it is capable of measuring damping, performs time-dependent measurements, and continuously sweeps the frequency for internal consistency checks. The block diagram is shown in Fig. 4; the grounded film is either driven electrostatically by a drive electrode connected to an external voltage amplifier that produces a 12V RMS oscillating voltage or by an exciting coil (not shown) connected to an adjustable 10–200 mA current source. A receiver electrode connected to a dc biased (130 V) preamp forms a small charged capacitor with the film; the biasing is required to produce charge flow when vibrations are induced.[11] The amplified signal, with its bias removed by a dc block at the preamp's output, is connected to a phase-locking unit (PLL). The PLL has a relatively wide capture range ($\sim \pm 15\%$) and locks to the resonant modes as tension is varied. The PLL also has outputs to a frequency counter (HP5316B) used for tension measurements, a frequency-to-voltage converter for time-

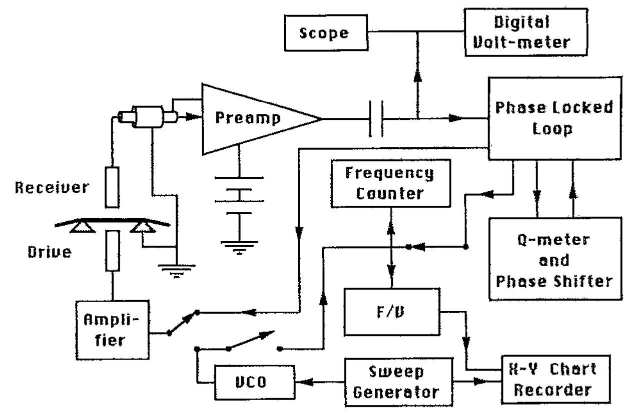

FIG. 4. Block diagram of the electronic system. VCO stands for "voltage controlled oscillator" and F/V for "frequency to voltage converter."

dependent resonant frequency tracking measurements, and a Q meter used to determine the damping in the film.

B. Preamplifier

Because the capacitance between the film and the receiver electrode is very small ($\sim$0.1 pf) a high input impedance circuit is required to detect the resonant signal. Otherwise, loading effects will dominate and severely damp the film's vibrations. A simple combination of an FET and a bipolar transistor (shown in Fig. 5) is used to minimize loading effects.[13,14] The bipolar transistor (T2) acts as a follower and ac bootstraps the input resistor RB which increases its impedance by several orders of magnitude ($\sim$100–1000) and thereby proportionately reduces its loading effects. The bootstrapping is accomplished by the capacitor CB that connects T2's emitter output to the junction between RA and RB. To eliminate other loading effects, the input cable capacitance of the receiver electrode and the capacitance of the FET's gate drain are also bootstrapped, the latter through capacitance CA.

The signal from T2's emitter output is ac coupled to a gain op-amp whose output is connected to a double-pole single-throw switch SW1. Normally SW1 is operated in the position shown in the figure (the two op-amps U2 and U3 in the lower half of the figure are bypassed). Since for damping measurements more gain is required, the switch is toggled to route the signal through U2 and U3; to be first filtered and then amplified. U2 is a unity gain second-order 1000-Hz high pass filter; this op-amp removes the low-frequency disturbances from the signal. The next op-amp U3 provides 50X gain for damping measurements.

It is important to note that the entire circuit is dc biased (130 V) to charge the receiver electrode and the film. The dc bias is removed from the signal by a blocking capacitor (see in Fig. 4).

C. Phase-locked loop (PLL)

Although the detection system described in the previous section detects the resonance of the membrane at its resonant frequencies (f) the resonances can be driven at

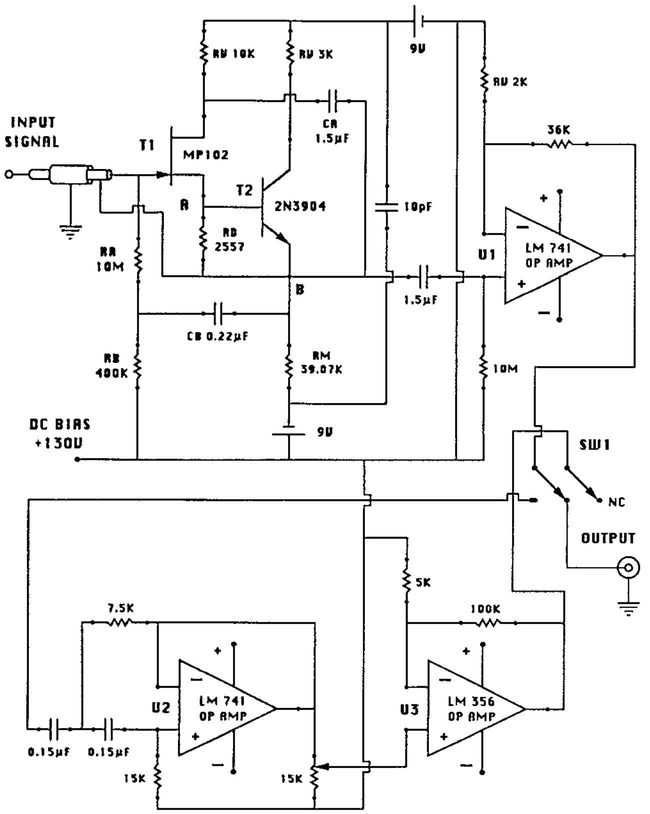

FIG. 5. dc biased pre-amp. The switch SW has two settings. In position 1 the op-amps U2 and U3 are bypassed. In position 2, U2 and U3 provide sufficient gain to enable damping measurements to be performed. All supply voltages are dc biased (differentially they are ±9 V).

either f of $f/2$. In order to avoid any parasitic pickup by the detector it is usually preferable to operate in the $f/2$ driving mode. However, since in some cases the driving efficiency can be greatly enhanced by operating at f, our system is capable of operating at both f and $f/2$. For each mode an independent PLL chip (NE 565) is used [Fig. 6(a)] and single pole double-throw toggle switch (SW2) selects the desired frequency. SW2 toggles between triangular outputs of U1 and U3 derived from their pins at positions number 9. At $f(f/2)$ the switch connects U1 (U3) to U5. U5 is a buffer amplifier.

A careful examination reveals that both PLL chips have similar circuitries except for their inputs derived from different sources. U1 is directly driven by the preamp. But U3 is driven by a JK flip flop (U2) whose function is to supply a waveform whose frequency is half the signal frequency (U2 is driven by U1; pins number 4 and 5). CF and CT are filtering and timing capacitors, respectively. RT's are timing resistors whose functions will be described in more detail below. The triangular waveforms from the PLL chips (NE565's U1 and U3) are connected to a buffer amplifier through SW2. The output from U5 is amplified by U6 before converted into a sine wave by the circuit shown in Fig. 6(b). The sine wave output is routed through a phase-shifter (located on the next module; see Sec. III D below) from which the phase-shifted output is returned back and amplified by U7 [Fig. 6(c)]. The 5-K

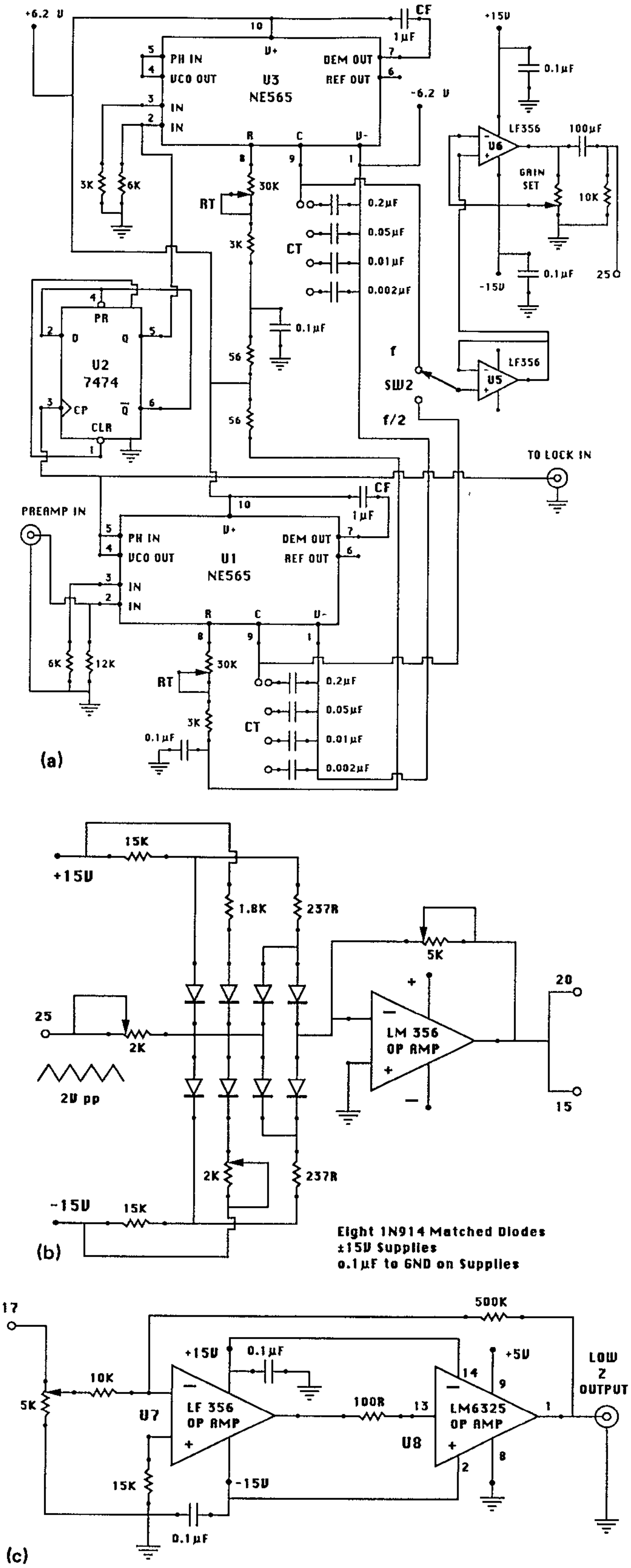

FIG. 6. (a) Phase-locked loop (PLL). The circuit operates in two different frequency modes; f and $f/2$. The tuning resistors (RTs) and capacitors (CTs) are externally adjustable. The triangular wave at pin 25 is connected to a sine-wave converter. (b) Sine-wave converter. The $2V_{pp}$ triangular wave at pin 25 comes from the PLL chips. The op-amp LM356 is a buffer amplifier and transforms current into voltage. (c) Gain and buffer amplifiers.

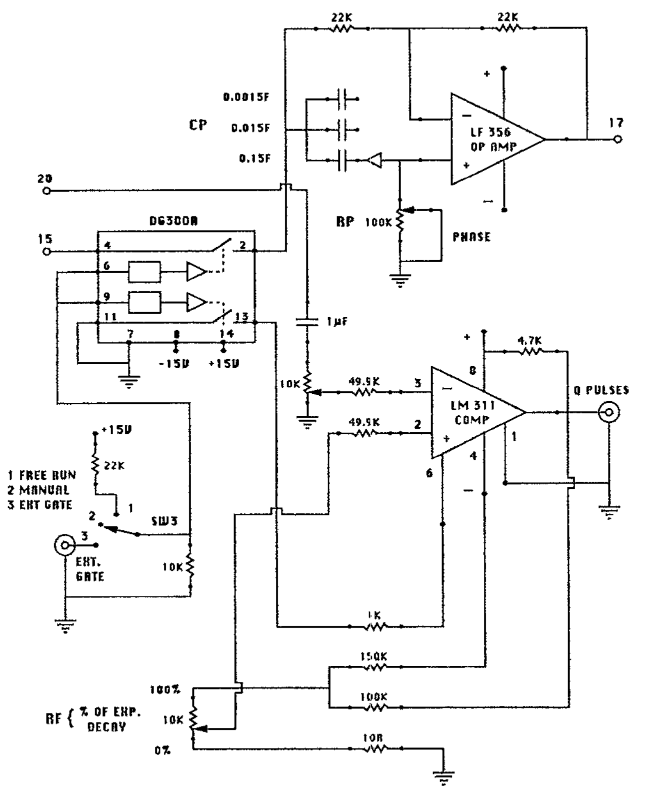

FIG. 7. Q meter and phase shifter. This circuit operates in three different modes, i.e., free run, manual, and external. All supply voltages are provided with 0.1-μF capacitors to ground (not shown). RF, RP, and CP are externally adjustable.

potentiometer provides an externally adjustable gain; so as to control the low impedance output of the high current amplifier U8 used when film is magnetically driven. When the film is electrostatically driven the output of U8 is amplified by a voltage amplifier (HP467A).

To operate the PLL, the toggle switch SW3 (see Fig. 7) is manually turned into position 1, "free run mode," which causes the digital switch DG300A to close pins 2 and 4. The TTL switches in DG300A (see Fig. 7) are open when logic levels at pins 6 and 9 are zero and closed when logic levels are raised to one. The sine-wave signal from pin 2 is phase shifted by an op-amp (LF356). Three capacitors (CPs) are provided to select the coarse ranges of the desired phase shift with a 100-kΩ ten turn helipot (RP) available for fine phase adjustments.

To lock the loop it is necessary to vibrate the film close to its resonant frequency. To accomplish this, SW2 is set either on f or $f/2$ depending on the drive mechanism, and a timing capacitor [CT, see Fig. 6(a)] is chosen to operate the loop in the desired frequency range. While monitoring the output of the preamp (see Fig. 4) on an oscilloscope the timing resistor RT is slowly varied and the loop frequency is monitored by a counter (HP5316B). It is important to properly adjust the coarse and fine phase adjustments (CP and RP, respectively, see Fig. 7) to maximize the signal. When locking occurs the loop frequency no longer varies. The procedure outlined above sometimes requires a number of attempts to succeed. With the loop locked RT and RP are further adjusted to maximize the received signal. The PLL remains locked if the film tension is varied slowly by the micrometer M. The lock can eventually be broken if the film's resonant frequency falls outside the capture range of the loop ($\pm$%15), but this can be avoided by readjusting RT and RP each time tension is varied.

D. Q meter

The Q meter is used to study the damping of the oscillations and its design is simpler than those described in the literature.[15-17] The Q meter itself consists of a voltage comparator (LM311) that generates counting pulses to determine Q (quality factor). It also incorporates the PLL to initiate the damped oscillations.

If a film is set into resonance and then the drive voltage is turned off the film oscillations decay exponentially such that

$$Q^{-1}=\frac{1}{n\pi}\ln\frac{V_0}{V_n}, \tag{2}$$

where V_0 and V_n are the amplitudes of the starting and nth oscillation. From Eq. 2 it is clear that the quantity of interest is the number of damped oscillations that occur above a fixed reference voltage level (V_n). The reference voltage is set by a 10-kΩ ten turn precision potentiometer RF (Fig. 7). The reference voltage is 0 V when RF's center tap is at ground, and is 1.000 V at the 100% position with a linear variation between the two limits.

To measure the Q the PLL loop is first locked as described above (see Sec. III C). Note that for damping measurements the pre-amp must be operated in its high gain mode. With the lock established, the PLL components (RT and RP) are adjusted to maximize the signal. A digital voltmeter gives the signal amplitude [V_0 in Eq. (2)] at the preamp output (see Fig. 4), and is recorded for future use. Now RF is adjusted to set the desired reference voltage V_n. The PLL switch SW3 is turned into its mid position (manual mode; position number 2) which causes both switches in DG300A to open. The opening of the upper switch disconnects the drive voltage and initiates the damped oscillations. The lower switch strobes the comparator and initiates counting pulses for summation by a frequency counter (HP5316B). The counting terminates when the amplitude of the signal falls below V_n.

With switch SW3 in position 3 (external gate), the digital switch DG300A may be triggered by external rectangular pulses thus allowing n to be measured repeatedly. For each rectangular pulse the above functions are repeated; i.e., the PLL is locked and then opened to count the number of damped vibrations. With this procedure a large number of measurements may be averaged to reduce statistical errors.

E. Auxiliary units (VCO and F/V)

The auxiliary units consist of the VCO (voltage controlled oscillator, Fig. 8), and F/V (frequency to voltage

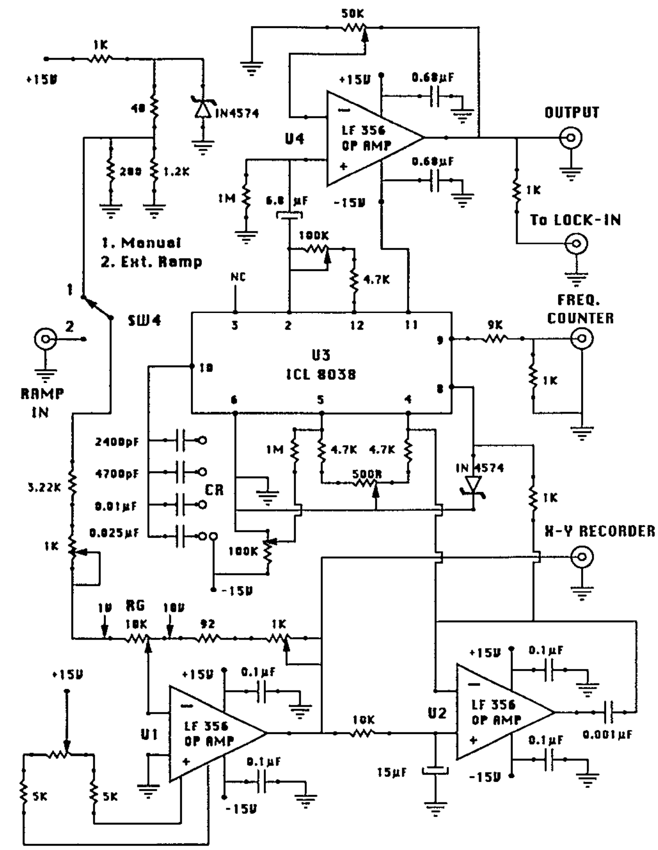

FIG. 8. Voltage controlled oscillator circuit. CR and RG are externally adjustable.

converter, Fig. 9). The VCO chip ICL8038 generates a voltage controlled frequency that sweeps the film through its resonant modes to verify if resonances correspond to the modes of a uniformly tensioned membrane. In conjunction with the VCO, a lock-in amplifier, connected to the output of the pre-amp, is used to chart the resonances on an X-Y recorder.

In Fig. 8 U1 is a variable gain input amplifier. The 10-kΩ ten turn precision helipot RG sets the ratio of the output frequency (Δf) to the input voltage (ΔV). The VCO circuit can be swept either manually or by an external ramp voltage as selected by the switch SW4. In the manual mode RG varies the output frequency whereas in the external mode it sets the scale of the output frequency. U2's function is for linearization purposes (when the VCO operates at extreme ends of its frequency range). Four capacitors connected to pin 10 of the VCO chip (values indicated next to each) are used to select the appropriate frequency range. The VCO output at pin 2 is buffered by an op-amp U4.

The purpose of F/V is to track time-dependent changes in the PLL frequency, for instance; to study changes produced by temperature, irradiation, thin-film overlayer deposition, etc. Chip AD453 is employed (see Fig. 9) for frequency to voltage conversion with the input signal connected to pin 2 through a switch. In the internal mode this switch connects the circuit to the PLL. A 100-kΩ feed-back helipot RS between pins 4 and 8 sets the ratio of $\Delta f/\Delta V$ (the voltage V varies between 0 and 10 V). A 50-kΩ ten turn helipot (whose center tap is connected to pin 1) sets the dc output voltage to zero at the desired frequency. The output voltage at pin 8, connects to the noninverting input of a low drift op-amp whose inverting input, connected to a 5.000 V supply, functions as a summation amplifier to remove dc drifts.

IV. RESULTS AND DISCUSSION

A number of films composed of different elements (Ta, Ni, Cu, Nb, and Ag), and prepared by a variety of techniques (rolling, electrodeposition, sputtering, and evapora-

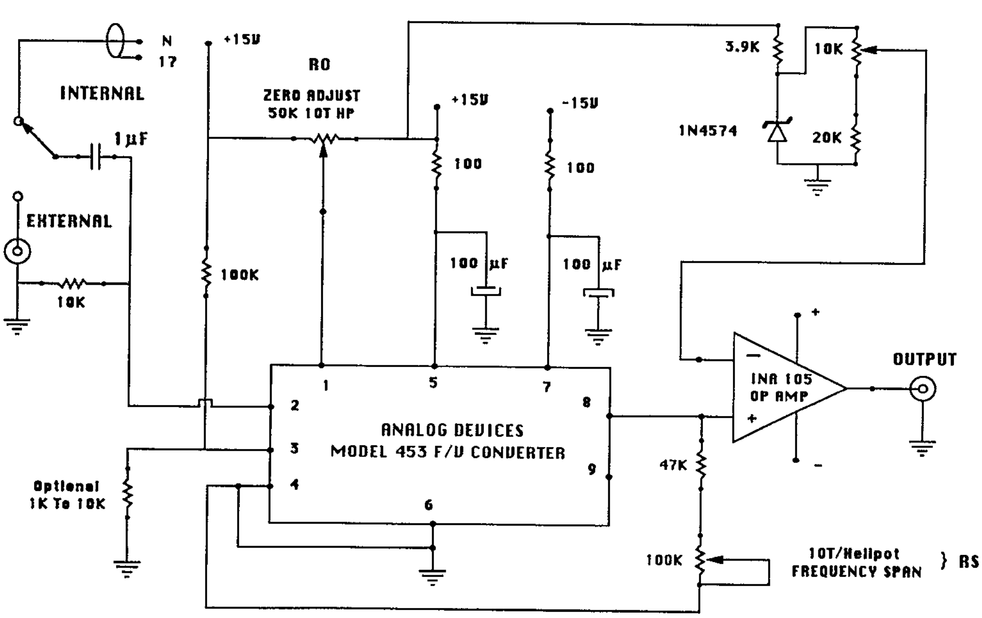

FIG. 9. Frequency-to-voltage converter. RO and RS are externally adjustable.

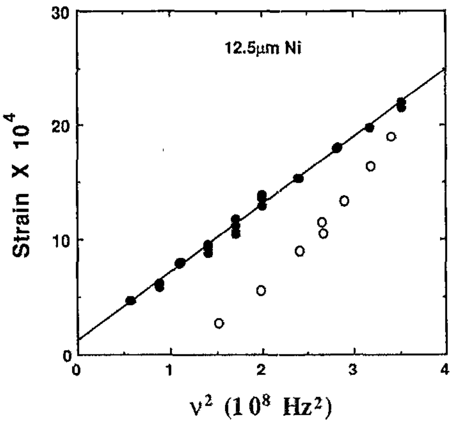

FIG. 10. Strain vs ν^2 of an electrodeposited [100]-oriented nickel film. Tension is reduced (increased) for filled (unfilled) circles. For filled circles $\Delta(\text{strain})/\Delta\nu^2 = 5.938\times10^4\ \text{s}^2$ and $Y_B = 0.226$ TPa.

tion) have been tested. Figures 10, 11, and 12 show the strain versus the square of the resonant frequency ν^2, for [100] nickel (electrodeposited), [110] niobium (sputtered), and [111] silver (evaporated) films. In each plot the unfilled (filled) circles correspond to values of increasing (decreasing) stress. The use of Eq. (1) and the slope obtained from a least squares fit to the filled circles gives the biaxial modulus. the Y_B's so determined are in good agreement with those calculated from the bulk crystalline elastic constants C_{ij}'s for the particular crystallographic textures (measured by x-rays) of each film.[18]

As stress is increased the data for Ni (Fig. 10) are nonlinear indicating some degree of plasticity. When stress is released the curves are linear and yield a biaxial modulus (Y_B) equal to 0.226 TPa in good agreement with the calculated value of 0.222 TPa.[18] The niobium film (Fig. 11) shows no plastic behavior within experimental error, and is linear and reversible in both directions of stress. The Y_B is 0.164 TPa compared to the expected value of 0.167 TPa.[18]

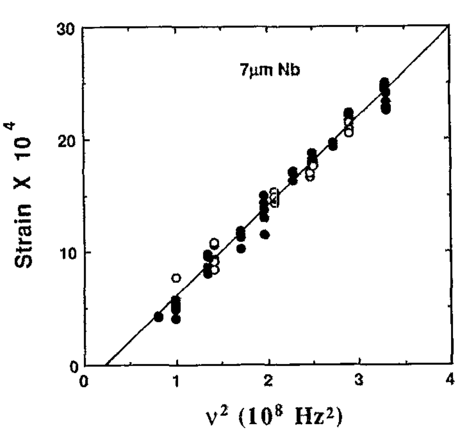

FIG. 11. Strain vs ν^2 of a sputtered [110]-oriented niobium film. Tension is reduced (increased) for filled (unfilled) circles. For filled circles $\Delta(\text{strain})/\Delta\nu^2 = 8.135\times10^4\ \text{s}^2$ and $Y_B = 0.164$ TPa.

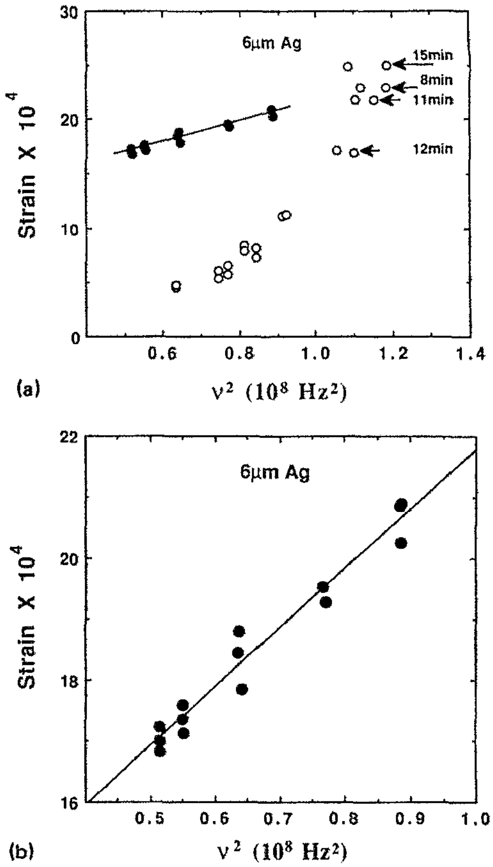

FIG. 12. (a) Strain vs ν^2 of an *e*-beam evaporated [111]-oriented silver film. Tension is reduced (increased) for filled (unfilled) circles. The arrows indicate relaxation of stress due to film's plasticity with the time interval of each measurement shown next to each arrow. For filled circles $\Delta(\text{strain})/\Delta\nu^2 = 9.73\times10^4\ \text{s}^2$ and $Y_B = 0.167$ TPa. The calculated Y_B for silver is 0.1734 TPa. (b) Strain vs ν^2 for decreasing tension corresponding to the stable points shown in (a).

Silver exhibits even larger plastic deformations than Ni, and it undergoes increasingly rapid stress relaxation at frequencies above $\sim$10 kHz which precludes stable measurements at higher frequencies [Fig. 12(a)]. The arrows show the points affected by the rapid relaxation of stress for the indicated times. Note that since the strain remains constant, whereas the stress (ν^2) is a function of time the changes in stress must be induced by the film's own intrinsic response.

The stress relaxation rate becomes so large above $\nu^2\sim10^8\ \text{Hz}^2$ ($\nu\sim10$ kHz) that no meaningful increase in stress results with increasing the strain. At this frequency ($\nu = 10\pm0.2$ kHz) the film strains freely indicating the "flow stress" of the film has been reached. On releasing the strain in small steps, the stress is found to be stable below $\nu^2\sim0.9\times10^8\ \text{Hz}^2$. In contrast to nickel and niobium, for silver it is not possible to measure its elastic response over a wide frequency range. In spite of this, the data (the stable ones obtained at low stresses) yield a biaxial modulus in good agreement with the expected value [Fig. 12(b)]. The measured value is 0.167 TPa, and the calculated one is 0.1734 TPa.

Although for each film we test to ensure the validity of treating it as a membrane, it is important to estimate how much the plate elasticity might affect the resonant frequency. The measured frequency[18] is given to a good approximation by the following expression:

$$\nu_0^2 = 0.1478 \frac{T}{a^2\rho} + 0.23 \frac{Eh^2}{a^4\rho(1-\eta^2)}, \tag{3}$$

where h is the film thickness, E is the Young's modulus, and η is the Poisson's ratio. The above equation reveals that Y_B [measured from the slope of $\Delta(\text{strain})/\Delta\nu^2$] will not be affected by the film's plate elasticity. It may appear from Eq. (2) that additional information could be obtained from the intercept of strain versus ν^2 data. Experimentally this is not possible due to uncertainties in the orgin for strain which is ill-defined because of any initial warping and/or plastic deformation.

Three sources contribute to a film's damping; hydrodynamic (by air), mechanical (losses at clamping boundary), and intrinsic (due to losses in the film material itself). Hydrodynamic damping can be reduced to negligibly small values if the film vibrates in a vacuum better than 10^{-5} Torr.[11] Although the measurements presented here were performed in a vacuum of 10^{-3} Torr the Q's were sufficiently high ($\sim 10^4$) for biaxial modulus measurements. The losses at the film clamping boundary are harder to estimate. However, preliminary studies for a number of films at pressures of $\sim 10^{-5}$ Torr yield Q's close to that of their intrinsic bulk values which indicates that the knife-edge support acts as an efficient clamping boundary which dissipates a negligibly small portion of the vibrational energy. Since here we only discuss functional aspects of our device more detailed studies on damping will be published elsewhere.

In conclusion, we have successfully developed and tested a compact apparatus for mechanical measurements of metal films that utilizes their membrane properties. The purpose of its construction was to enable us to reliably measure the biaxial modulus of metal films. In this respect it offers certain advantages over its counterparts (such as bulge tester) since the films are configured to obey more closely the model employed in the data analysis.[9,19] The results are less susceptible to uncertainties caused by handling of thin metal films, and since only membrane properties are used, explicit knowledge of film thickness is not required.

ACKNOWLEDGMENTS

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