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MASTER

THIN FILM EVAPORATION RATE MONITOR

by

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RELEASED FOR ANNOUNCEMENT
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ABSTRACT

This report describes instrumentation which will permit constant monitoring of the rate of metal evaporation, as well as measurement of the final thickness deposited upon a substrate.

SUMMARY AND CONCLUSIONS

1. Satisfactory operation of the evaporation rate monitor was obtained throughout the thickness range of 25 to 10,000 angstroms. The thickness of the film could be determined from the equation -

$$t = K\Delta F \quad \text{Eq (1)}$$

where t is the thickness of the film coating deposited, K is a constant of proportionality that is dependent upon the original frequency of the crystal and the way in which the crystal is cut (see Appendix A on different cuts for quartz crystals), and ΔF is the frequency change experienced by the crystal due to the film deposited upon its surface. This technique is not recommended for film coatings which require rotating contact leads such as might be used when a sample is supported on a revolving platform. The high frequency used in this system (27.225 mc) is too high for the distributed capacitances existing near the monitoring crystal, therefore, a sustained natural oscillation is not possible due to loading of the circuit.

2. This device has the advantage of good accuracy and rapid response at both the low (25Å) and high (10,000Å) ends of its range. Metal films thicker than 15,000Å deviate too much from Eq 1, and should not be measured unless proper correction factors are used to compensate for non-linear response of the crystal at these thicknesses.
3. Outgassing difficulties on the first circuits to be designed were serious due to inclusion of rubber and neoprene covered coaxial cable in the vacuum system. This problem was solved by using an open transmission line between the output of the amplifier circuit and an octal feed-through connector on the wall of the vacuum chamber, or by using Teflon (TFE) coated lead wire.

4. The range of the crystal exceeds the upper limit of the frequency counter (100 kc). When the frequency displayed by the frequency counter reaches 100 kc, the monitoring crystal is covered and used as a reference crystal. The reference crystal then is exposed to the metallic evaporation, thus becoming a monitoring crystal. The frequency change, in this case, will be a descending function and will have the same limits of accuracy as in the original conditions.
5. Accuracies in the range of 1 percent at 50 kc can be realized with this monitoring system. The lower usable limit is determined by circuit noise which averages about 16 cps.

INTRODUCTION

Vacuum evaporation processes usually require removal of a coated substrate from the vacuum chamber so that the coating thickness can be measured by methods such as multiple beam interferometry or weight gain. Accurate control of film thicknesses is difficult to attain, and the time required to produce each coating is excessive. A device was needed that could monitor the thickness of the film at all times while deposition was taking place. An instrument with such a capability would allow a speedier operation and a higher degree of accuracy. Repeated evacuations of the vacuum chamber would be eliminated since the operator would know more precisely when the desired film thickness has been obtained.

This report describes the construction of an evaporation rate monitor consisting of crystal controlled oscillators, a mixing circuit, amplifier circuit, and power supply to fulfill our requirements. A block diagram of the circuit is shown in Figure 1.

Figure 1. MONITORING CRYSTAL BLOCK DIAGRAM

Transistorized circuitry was used throughout in order to obtain minimum heat dissipation in a small package. A printed circuit card was designed for the amplifier stage to speed construction of this circuit. Positioning of the monitoring crystal is also discussed since mounting of this unit becomes quite important for proper functioning of the circuit. Schematic diagrams of the various circuits are included in this report.

DISCUSSION

A. THEORY OF OPERATION

The relationship between the frequency of oscillation of a quartz crystal and its thickness is well known (Appendix A). Quartz crystals can be of various types according to the way they are cut. Various cuts affect the frequency of oscillation, stability, and have different temperature coefficients. This oscillator makes use of an AT-cut crystal, which is inherently stable over a wide temperature range. The self-oscillation of a crystal is dependent upon the thickness of the quartz, and it is this property which is used in this system to measure the thickness of the deposited metal. The greater the amount of metal evaporated upon the quartz crystal, the greater the thickness of the crystal and the lower its frequency of oscillation (F_1).

A second crystal with the same approximate resonant frequency as the monitoring crystal is used as a reference so that by comparing both signals, the frequency change (ΔF) of the monitoring crystal can be more easily detected and fed into a readout device.

The monitoring frequency (F_1) and the reference frequency (F_2) are fed into a mixer circuit where they are mixed or heterodyned so that at the output there will be several frequencies: the fundamental frequency F_1 or F_2 (whichever is greater), the difference frequency $F_2 - F_1$ which constitutes the pulsating frequency of interest, and the sum frequency $F_2 + F_1$. The mixer circuit is a tuned amplifier whose output consists of the higher reference frequency F_2 and a pulsating signal of frequency $F_2 - F_1$. This

difference frequency will be almost zero cps before any metal is evaporated on the monitoring crystal provided the natural frequency of oscillation of both crystals is equal ($F_1 = F_2$). The frequency change (ΔF) of the monitoring crystal, after being exposed to some metal deposition, will cause the frequency F_1 to decrease by a proportional amount. Therefore, the resulting frequency will be:

$$F_1 - \Delta F$$

Since the reference frequency suffered no change, the inputs to the mixer circuit will be $F_1 - \Delta F$ at one input and F_2 at the other input terminal. However, the combining of two waves of slightly different frequencies results in a wave having an envelope that pulsates in amplitude at the difference frequency of the component waves. Therefore the output of the mixer will consist of the frequencies F_2 , $F_2 - (F_1 - \Delta F)$ and $F_2 + (F_1 - \Delta F)$. Assuming that the original frequencies F_1 and F_2 were equal, the signal delivered by the mixer will consist of two components; the frequency F_2 and the pulsating signal ΔF which is equivalent to the frequency change effected upon F_1 after having been exposed to some metal deposition. The frequency sum $F_1 + F_2 - \Delta F$ is high enough in frequency to go undetected, and therefore it is not considered in this analysis.

The frequency F_2 along with its pulsating component ΔF are then fed into the amplifier demodulator circuit (Figure 2) where the signal is rectified and additional amplification provided. The high frequency component is then removed in the last stage of amplification thus leaving a pulsating component which lies within the range of the frequency counter. This output frequency ΔF can then be correlated to the film thickness deposited upon the substrate.

Since the frequency ΔF covers a range from zero to some audible frequency such as 15,000 cps, an audio tone can be used as a second monitor device in conjunction with a frequency counter. An audio amplifier limits the amount of metal which can be deposited upon the monitoring crystal, since, for too great a thickness " t ", the resulting frequency ΔF will be greater than 15,000 cps which

Figure 2. AMPLIFIER CIRCUIT AND DEMODULATOR DIAGRAM

is outside of the audible range. The original resonant frequency of these crystals is 27.225 mc. Therefore, the slightest foreign particle upon the quartz will cause a considerable change in cycles per second, thus making this type of measurement extremely sensitive. (The frequency of oscillation "f" is related to the crystal thickness "t" according to the following expression:

$$f = \frac{66.2}{t}$$

where f is the frequency in mc/sec. and t is the thickness in mils.)

In use, the monitor crystal is positioned immediately adjacent to the substrate to insure it will be coated with the same proportional amount of metal as that deposited on the substrate. A 1/4-inch diameter hole is drilled in the monitor crystal case so that the evaporant metal can be deposited on the quartz crystal. The quartz crystal mounting plate must be carefully unsoldered and removed before this hole is drilled in the case. The crystal cover can then be replaced and soldered back if desired. The reference

crystal is enclosed in a small container housing the mixer and amplifier circuits and, therefore, it is not exposed to any metallic evaporation which may cause a frequency change. This container is also mounted adjacent to the substrate to achieve a constant monitor/reference temperature.

Calibration of this instrumentation was accomplished by depositing aluminum films of varying thicknesses upon several 3 by 1 inch glass slides for interferometer measurement. The frequency changes caused by these varying thicknesses were recorded, and later correlated with multiple beam interferometer results. Subsequent film thicknesses can be determined from the number of cycles in frequency change necessary for any required thickness. A shutter mechanism located between the source and the substrate will then allow the evaporation to be stopped when a preset thickness is reached.

Figure 3. CRYSTAL CONTROLLED OSCILLATOR DIAGRAM

1. Oscillator (see Figure 3)

The oscillator circuit is crystal controlled and uses a high-speed triode switching transistor Type 2N711. The crystal used is of the citizens band AT-cut type so that frequency drift can be kept to a minimum as crystal temperature varies. The frequency of the quartz crystal is stamped on the outside of the case as

being 27.225 mc; however, since this is a third overtone crystal, its fundamental frequency is actually 9.075 mc. D.C. voltage to drive the oscillator circuits is derived from a Hewlett-Packard Model 721A transistorized power supply. A voltage dropping resistor in conjunction with a variable potentiometer provides a drop in voltage from 20 volts down to 5 volts required for proper operation of the oscillator (see Figure 4).

Figure 4. CURRENT MONITOR AND OSCILLATORS POWER SOURCE

A 0-10 ma. milliammeter is used in series with the oscillator circuits to provide some means of checking the performance of these units. Voltage regulation for this circuit is provided by a IN468 zener diode rated at 4.9 volts and 200 milliwatts. Excellent voltage stability is obtained from this zener diode throughout the current range of the oscillator circuits which can be adjusted for 6-8 ma. for best performance. Since the quartz crystal is very thin for this frequency of oscillation (9.075 mc) excessive current through the oscillator circuits can damage the quartz easily; therefore, the current should be held to a low value (10 ma. or less). A 0.01 mfd capacitor shunting the zener diode is used to decouple high frequency voltages from the power supply and should not be omitted.

The monitor crystal is connected to the oscillator circuit through open transmission lines made of 3-inch long pieces of No. 10 copper wire connected to a crystal socket. Extension of the crystal socket permitted a cooler operation of the oscillator circuit by increasing its distance from the source of heat, therefore, component failure (mainly transistors) was cut to a minimum.

The reference crystal may be mounted in several positions depending on its function. These positions include the following:

- Adjacent to the substrate and covered with a shutter in instances where the reference and monitor crystals are used interchangeably to measure thickness.
- Adjacent to the substrate and completely enclosed when the only function is to achieve a constant temperature equal to the monitor crystal temperature.
- Outside the system on the mixer input when optimum accuracy is not required.

Necessary trouble shooting can become a simple matter merely by substituting a monitor oscillator outside of the vacuum chamber and determining if the source of trouble is in the monitoring unit. The cabling required to transfer the signal from the feed-through connector to the mixer circuit consists of several feet of RG-59/U coaxial cable and a separate insulated wire for the D.C. voltage. It is suggested that excessive lengths of wire be avoided so that neither the D.C. voltage nor the high frequency signal will be unduly attenuated.

2. Mixer Circuit (see Figure 5)

This circuit is used to mix the monitor and reference frequencies to produce a beat frequency. The circuit consists of a tuned frequency amplifier whose main purpose is to let through any frequency near 9 mc and at the same time attenuate all other signals on a different frequency.

NOTE: T₁ & T₂ are I-F Transformers tuned to 10.7 Mc Miller 1463 interstage.

Figure 5. MIXER CIRCUIT DIAGRAM

Transformers T₁ and T₂ were originally tuned to 10.7 mc; however, by slightly detuning their core slugs, they can be made resonant to 9 mc which is the approximate frequency output of the crystal oscillators. The 30 pico-farad air type variable capacitors adjust the amplitude of the incoming signals so that a symmetrical output signal may be obtained. It also compensates for small differences in stage gain and circuit elements.

Power to the mixer stage is derived from the Hewlett-Packard low-voltage supply whose output is adjusted to +20 volts.

3. Amplifier Demodulator Circuit (see Figure 6)

The output signal of the mixer stage requires additional amplification before it is fed into a frequency counter. The counter used in this system (Figure 6) was a Beckman EPUT meter Model 5210 DFR whose maximum sensitivity is 0.25 volts; therefore, the output signal from the mixer must have an amplitude greater than 0.25 volts so that the frequency counter may be able to detect this signal. Operation of the counter near its maximum sensitivity

resulted in erroneous operation, therefore, the mixer output was amplified to improve the counting accuracy of the instrument.

Figure 6. COMPLETE CRYSTAL MONITOR UNIT

The amplifier circuit consists of 3-transistor stages capable of a frequency bandpass from 100 cps to 100 kc. The output signal to the counter has an amplitude of 12 vpp. and, therefore, there is sufficient drive voltage for most counter requirements. Power to this amplifier is also derived from the Hewlett-Packard supply just as in the case of the mixer circuit. There may be occasions when the cost of a high-frequency counter cannot be justified; in that case, a less expensive rate meter can be used providing that a small decrease in accuracy can be tolerated.

The complete unit as originally designed is shown in Figure 6. The monitor crystal and oscillator circuit are labeled Items 1 and 2. Notice the distance existing between the crystal socket and the small package enclosing the oscillator circuit. Sleeves of aluminum oxide surrounded the open transmission line to provide a certain degree of insulation. The physical separation of the quartz crystal from the oscillator enclosure provided additional isolation for the amount of heat radiated. Positioning

of the crystal monitor in this manner greatly reduced maintenance of the oscillator circuitry.

Item 3 in Figure 6 shows the amplifier circuit in its finished form. The various circuit boards can be constructed separately, or if desired they may be integrated into a small final package.

Item 4 in Figure 6 encloses a crystal oscillator circuit which can be used for trouble shooting purposes. Power (5 v D.C.) for this item is fed into the banana jack in front, and the output signal is received at a BNC connector in the rear. This equipment allows the circuit to be checked without the need to vent the vacuum system and remove the monitor circuit. The upper part of the cabinet shown in Figure 6 consists of a panel with the low-voltage power supply and the various connectors required to link the oscillators with the readout and the oscillator power source. Notice the screw-driver adjustment just below the oscillator current meter. This adjustment should be made so that the current on the meter ranges between 6 and 8 ma. with both oscillators in the circuit. Connection to the crystal oscillators is not critical and, therefore, cables can be interchanged. (Left or right connector) The oscillator power does not require a shielded cable and, therefore, any well insulated wire can be used. The signal from the mixer circuit reads "Beat Frequency Output," and it is this signal which is fed into the frequency counter for a digital display of the difference frequency ΔF . The counter has a built-in gate whose period or time interval is controlled by a knob labeled "Time-Seconds." This knob is adjusted so that the digital presentation of the frequency being counted may be easily read as the frequency decreases. (Lower frequency requires greater counting time.) This function, in essence, is that of a rate meter whose readout is the number of cycles per unit of time; the greater the "Time-Second" setting, the better the accuracy of the signal being displayed.

Figure 7 shows the mixer and amplifier circuit integrated into a single board. The reference crystal and its associated circuit is also mounted on this board so that the monitoring crystal package

can be kept as small as possible. The box enclosing all these circuits was a 4 by 5 by 6 inch "Minibox" which presented no heat problem whatsoever, because there was hardly any heat dissipated by the circuits.

Figure 7. INTERIOR VIEW OF AMPLIFIER
DEMODULATOR CHASSIS

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EQUIPMENT MODIFICATIONS

Several equipment modifications have been incorporated into this circuitry for specialized use. These include the following:

- a. Multiple monitor crystals - as many as 4 monitor crystals have been incorporated into a vacuum system to allow measurement of evaporation rate or film thickness. These monitors are used either at various distances from a single metal source or to measure several types of metal film thickness. For these applications, it is necessary to obtain a starting beat frequency for each monitor and subtract this value from the final frequency. The monitor and reference must be zeroed using the 2 crystals with minimum starting frequency. It is also necessary to have calibration curves for each metal specie being measured since each metal will produce a different frequency change per angstrom.

- b. Automatic shutter - an automatic shutter located between the evaporation source and substrate can be incorporated to stop deposition at a preset thickness (frequency - ΔF).
- c. Counters - counters manufactured by Hewlett-Packard were found to be less erratic than the Beckman EPUT units.

APPENDIX A

The AT-Cut Crystal

The property of a quartz crystal to be able to oscillate at some frequency is based on the piezo-electric effect. The phenomena consists of the following: If mechanical pressure is applied to a crystal between 2 of its faces, a difference of potential will be created on those 2 faces. The converse is also true; that is, if a difference of potential is applied between 2 faces of a quartz crystal the shape of the crystal will be physically altered. It is this characteristic which is utilized in a quartz crystal to control the frequency of an oscillator within certain limits.

A crystal has 3 axes or planes along which pressure may be applied. The X axis is parallel to a flat side of the crystal as shown in Figure 8-A. The Y axis is normal to the flat side of the crystal. Each Y axis is perpendicular to an X axis, thus in Figure 8-A, Y is perpendicular to X, Y' is perpendicular to X', and Y'' is perpendicular to X''. The Z or optical axis is a line drawn between the 2 apexes of the crystal as shown in Figure 8-B. The "X" and "Y" axes of a crystal are called the

Figure 8A.

Figure 8B.

ELECTRICAL AXES OF A CRYSTAL

electrical axes because if a difference of potential is applied at either axis, the crystal will contract along that axis, and expand along the other. The Z axis, however shows no electrical effect, and therefore will neither expand nor contract when a difference of potential is applied along this axis.

There are several methods of cutting a crystal to take advantage of its piezo-electric oscillations for frequency control. The X-cut crystal is one of the most common since its greatest advantage is that it oscillates at 2 distinct and well-separated frequencies. One of the frequencies is determined by the X axis, or thickness dimension, and the other by the Y, or length dimension. The frequency of oscillation of an X-cut crystal can be found from the formula

$$f = k/t$$

where f is the frequency in cycles/sec., k is a frequency constant for X-cut plates equal to 113×10^6 , and t is the thickness dimension in mils.

The Y-cut or 30-degree crystal is similar to the X-cut crystal except that the quartz slab is obtained from a slice which is parallel to an X axis instead of parallel to a Y axis as in the case of the X-cut crystal. Both of these type of cuts are temperature sensitive, and therefore require close temperature control if the frequency drift is to be kept to a minimum. To alleviate this problem the AT-cut crystal was devised. This type of cut is a compromise between the X and Y-cut crystals which exhibit a negative and a positive temperature coefficient respectively. Figures 9A and 9-B show the change in the cut used to produce the AT-cut crystal. Another advantage of the AT-cut crystal over the previous types is its power handling capability which becomes extremely critical as the frequency goes up.

There are other types of crystals used in industry but the X, Y, and AT are the most common in use. Persons interested in other types of cuts should consult literature on the matter such as J. R. Harrison, "Piezo-electric Resonance, and Oscillatory Phenomena with Flexural Vibrations in Quarts Plates." Proc. I.R.E., Vol. 15, p. 1040, December, 1927.

Figure 9A. Y-Cut
Crystal Slab

Figure 9B. At-Cut
Crystal Slab

Figure 9. COMPARISON BETWEEN THE Y AND THE AT CUTS

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