Increasing the Accuracy of Quartz Crystal Thin Film Thickness Sensors

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Quartz crystal thin film sensors are routinely employed in the fabrication of thin film electronic devices such as OLED's, LCD's and Integrated Circuits. And while known for their ability to detect films as thin as one Angstrom and, when properly optimized, reliable enough to insure process repeatability, crystal derived thickness readings are often taken as indisputable measurements by process engineers and scientists. Unfortunately, this blind faith can have major negative consequences in the manufacture of sophisticated thin film products.

The measurement of film thickness was pioneered by G. Sauerbrey in the early 1960's. Using a resonating quartz disk, Sauerbrey showed the relationship between the frequency of vibration (resonance frequency) and the mass, or corresponding thickness, of material deposited on its surface. This relationship was refined in later years by C. Lu to account for the acoustic properties of the film. In its current form, the equation used to calculate film thickness is defined as:

$$\rho_f t_f = (\rho_q v_q / 2\pi Z f_c) \tan^{-1}\{Z \tan [\pi(f_q - f_c)/f_q]\}$$

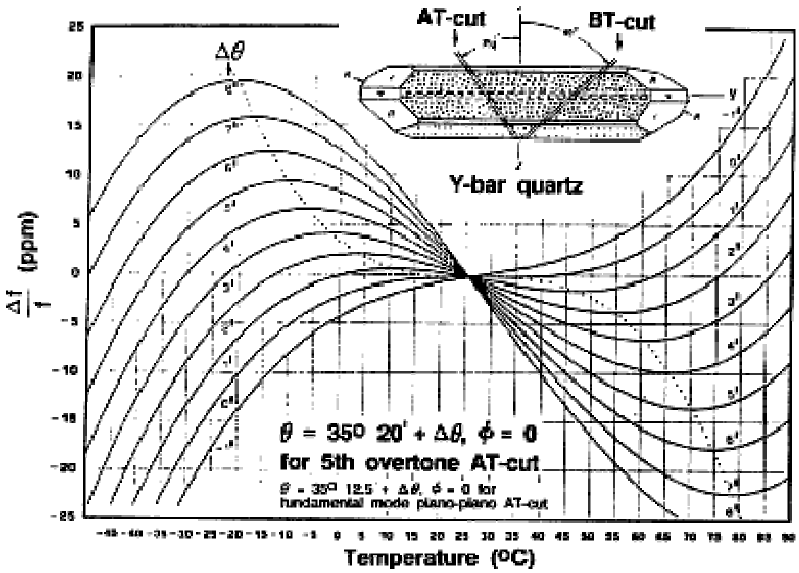
Where:

ρ_f	=	density of film measured
t_f	=	thickness of film
ρ_q	=	density of quartz
v_q	=	velocity of acoustic wave through quartz
Z	=	acoustic impedance factor
f_q	=	frequency of uncoated quartz
f_c	=	frequency of coated quartz

In a typical deposition run, the user programs the crystal controller with the density of the film being monitored, the acoustic impedance value (applicable only to thick films) and the tooling factor, a geometrical correlation of the deposition source to crystal and source to substrate distances. The primary assumption made is that the film density is known, and that it is not dependent on deposition conditions. For a given process configuration, the film density can be calculated exactly by comparing the crystal thickness with a secondary measurement obtained from profilometry, for example. If not, a significant error can be introduced to the film thickness calculation.

The greatest source of error to film thickness measurement arises, however, from factors not accounted for in a typical deposition. Although the quartz crystal resonance frequency has been shown to be sensitive to mass deposition, it also is sensitive to temperature, stress in the growing film, and adhesion of the film to quartz, among other factors. The temperature dependence is so great that quartz has been used as a thermometer.

To minimize temperature dependence, quartz crystals are cut from a quartz bar at a specific orientation angle. The angle most commonly chosen is referred to as the AT cut, 35° 15' from the Z or optic axis of the quartz bar. This cut has a near zero frequency-temperature dependence at 20°C. For a change of a few degrees in either direction, the quartz resonator will not exhibit any frequency shift. This implies that during a deposition run, the film thickness monitor will not interpret a slight warm-up of the crystal as additional mass deposited.



To further minimize temperature related frequency changes, quartz sensors are placed in water-cooled housings, with the cooling water set at 20°C. In theory, this configuration results in the frequency-temperature behavior being minimized or even eliminated. In practice, however, this is not realized and worse, the user is not even aware that it is a factor.

As sophisticated as the electronics have become for quartz crystal thin film measurement, in no case does the mention of crystal sensor temperature make its way into the operation manuals, except to the extent that they specify crystals must be cooled to 20°C. No commercial instrument actually measures this temperature either, in the event that the user chooses to adhere to this specification. In actual operation, we have monitored uncooled crystals during routine thin film depositions and found they can exhibit temperature changes of at least 50 to 100 °C. For water-cooled sensors, the crystal can experience 20 °C swings during a typical coating cycle.

The significance of temperature shifts becomes critical when measuring films less than 300 Angstroms. From the frequency-temperature charts, it can be shown that a 20°C shift can amount to an erroneous thickness reading of 20 Angstroms or greater in the final thickness reading. This error can easily double if the deposition source emits large amounts of radiation. The sudden heating of the crystal has been shown to lead to frequency changes of 50-60 Hz instantaneously, corresponding to thickness errors of 25 to 30 Angstroms for low-density materials.

To compound matters, commercially available quartz crystals are cut in a range of angles, typically 35°15' +/- 3'. This 6' window for crystals introduces another source of error in thickness measurement. Under identical deposition conditions, the variation of frequency change with temperature between crystals at 35° 12' and 35°18' amounts to 10% or more.

Unless end users account for the quartz crystal temperature effects, they will be unable to guarantee that run to run reproducibility will be possible for their thin film product or process. Worse, successful devices developed under strict protocols in a research laboratory my not be reproducible in pilot or large scale manufacturing runs.