

## **The Future of Quartz Crystal Thin Film Monitoring**

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Since the early 1960's, quartz crystals have successfully been used to monitor thin film coating processes used in the fabrication of optical devices such as lenses, filters, reflectors and beam splitters. Although initially employed as an aid to optical monitors to provide deposition rate information, quartz crystal sensors rapidly became relied upon to absolutely indicate and control optical layer thickness in automated deposition systems.

As demands have increased on optical devices, so too have the complexity of the thin film structures. While an antireflection coating consisting of a single layer of magnesium fluoride may have been sufficient 20 years ago, current designs may call for a 24 layer stack of alternating refractive index films. With the advent of high-speed optical communications, this stack increases ten-fold, leading to filters comprised of up to 256 layers.

The manufacturing of these geometries requires the control and accuracy a quartz crystal provides. Unfortunately, the materials and deposition temperatures utilized in today's processing adversely affect the operation of the crystal sensor. In some cases the crystal will fail abruptly during a coating run, while in others inaccurate measurements can result without the production engineer's knowledge. Aside from the inconvenience these problems cause, they may lead to the inability to produce the devices desired.

New developments in the field of quartz crystal thin film sensors directly address these issues. From the introduction of new sensors that operate at elevated temperatures, to the refinement of existing quartz designs to account for the stresses and failure mechanisms caused by optical materials, we will outline how this critical process control element is catching up to the requirements for the fabrication of the next generation of optics.

### **Current Practice**

Quartz crystal thickness monitors may be one of the most misunderstood components of an optical thin film deposition system. Typically used in conjunction with an optical monitor, quartz sensors provide the process engineer with coating rate and total thickness data in real time, with Angstrom level resolution. When set-up and calibrated correctly, crystal sensors can be used to automatically control deposition sources, ensure repeatable and precise thin film coatings, and determine optical film properties dependent on deposition rate. When used incorrectly, these same crystals can drive even the most seasoned process engineer to wits' end.

Quartz sensors measure film thickness by monitoring a change in the frequency of vibration of a test crystal coated simultaneously with the process substrates. As illustrated in Figure 1, the crystal is contained in a water-cooled housing, mounted in a line of sight

position relative to the coating source (electron beam, thermal evaporation, sputtering, etc.). The substrates to be coated are positioned as close to the crystal as possible, ensuring that the amount of evaporant falling on the substrate and crystal are essentially identical. (If this is not the case, a geometrical correction, called the tooling factor, can be applied).

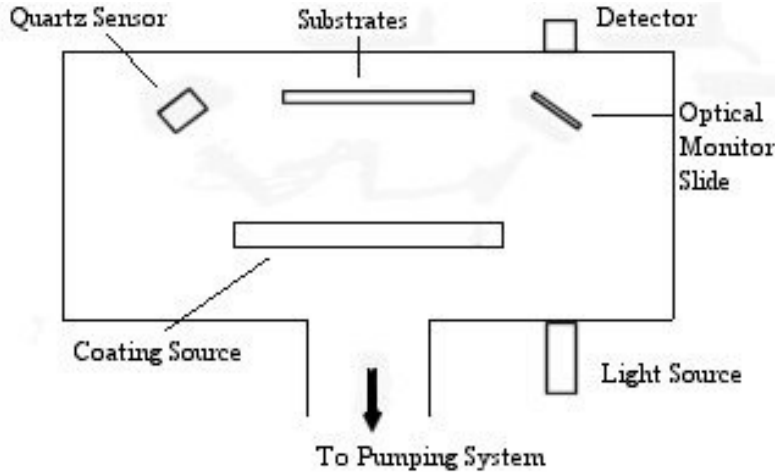


Figure 1: Vacuum Deposition System

The crystal is coupled to an electrical circuit that causes the crystal to vibrate at its natural (or series resonant) frequency, which for most commercial instruments is between 5 and 6 MHz. A corresponding microprocessor based control unit monitors and displays this frequency, and /or any derived quantities, continuously. As the source material coats the crystal during the deposition process, the resonant frequency decreases in a predictable fashion, based on the rate at which material arrives at the crystal, and its density. The frequency change is calculated several times per second, averaged, converted to a thickness value via an algorithm stored in the microprocessor and displayed as deposition rate, in Angstroms per second. The accumulated coating over time is also displayed, as the total thickness.

The sensitivity of the sensor is remarkable. A uniform coating of as little as 10 Angstroms of aluminum will cause a frequency change of 20 Hertz, easily measured by today's electronics. As the density of the film increases, the frequency shift per Angstrom increases, further increasing thickness resolution. Gold, at a density of  $19.3 \text{ g/cm}^3$ , decreases the resonant frequency 150 Hz for every 10 Angstroms deposited. Most monitors also report fractional Angstrom values, as a result of calculating frequency shifts per unit time.

The useful life of quartz is highly dependent on the thickness and type of coating monitored. If a low stress metal such as Aluminum is deposited, layers as thick as 500,000 to 1,000,000 Angstroms have been measured. At the other extreme, highly stressful dielectric films will reach less than 2 to 3,000 Angstroms before the crystal begins to malfunction.

## Shortcomings and Limitations

In the early days of quartz crystal thin film monitors, metallic films of copper, silver and gold were among the major materials deposited. These elements produced coatings of relatively low stress and were condensed on substrates routinely held at or near room temperature. Under these conditions, very accurate and reproducible determinations of film thickness and rate were achievable.

When the optics industry discovered quartz crystals, the attention shifted from metals to compounds transparent to visible light. Magnesium Fluoride, Silicon Dioxide, Titanium Dioxide and other dielectrics were the materials of choice. Unfortunately, these substances produced films with high intrinsic stresses and usually required high process or substrate temperatures. These were not welcome developments for quartz crystal monitoring.

The reasons can be traced to a fundamental property of quartz, piezoelectricity. If a bar of quartz is deformed or bent, it will develop a measurable voltage on opposite faces. Conversely, if a voltage is applied to a quartz bar, it will move or bend. By applying an alternating voltage to this bar of quartz, one can force it to vibrate in phase with the voltage in a process referred to as oscillation.

At a specific frequency of oscillation, quartz will vibrate with minimal resistance, much like a piece of fine glass crystal rings when struck. Early researchers used this natural resonance frequency as the basis for measuring film thickness. By added coatings to the crystal surface, or more precisely, “mass loading” it, they observed that the resonance frequency decreased linearly. If the coatings were removed, the resonance frequency increased.

To complicate matters though, if a quartz crystal is deformed while oscillating, as a result of thin film stresses or mechanical forces arising from the mounting holder, the resonant frequency will also change. If the deposition process conditions cause the crystal to be heated or cooled, a similar frequency shift occurs. Regardless of the origin, the frequency shift is indistinguishable from that caused by mass loading.

Additional resonant frequency changes can be caused by: 1) extraneous vibrations introduced through the mounting hardware, 2) amplitude variations in the voltage used to oscillate the crystal, 3) vibrational characteristics of the film being monitored (referred to as acoustic impedance), 4) adhesion failure of the monitored coating or the electrodes from the quartz surface, and 5) radio frequency interference in the monitoring circuit. These frequency shifts can be positive or negative, and can be cumulative. Worse yet, they can be random.

These unaccounted for effects can introduce large errors in thickness and rate. Uncontrolled temperature swings in quartz can result in thickness variations on the order of 50 Angstroms or more. Adhesion failure has resulted in 100-Angstrom spikes during

deposition. Extraneous vibrations, sometimes referred to as spurious modes, can produce changes in the tens of hundreds of Angstroms range. For precision optical components like DWDM filters, or optical- electronic devices such as organic light emitting diode displays (OLED's), these errors result in major yield loss.

The harsh conditions present during optical film coating have a deleterious effect on the operating life of the crystal as well. High stress coatings can deform the crystal to the point that it ceases oscillation, often without warning. Splatters or eruptions of material from the coating source can lead to the same failure. High-energy plasmas or glow discharges used for substrate cleaning can arc across the crystal surface, coupling into the electronics and causing severe electrical noise, obscuring the frequency signal. High temperature depositions can overheat the crystal, driving it past its operating limit.

Early crystal failure can be either a great inconvenience or an unmitigated disaster. In the case of 100 or more layer thin film stacks, simply venting the chamber to replace crystals is not an option, due to the undesirable effects of atmospheric gases on the film chemistry. For very thick films, used in laser power or infrared optics, short crystal life may lead to excessive run times as a result of the added time to cool and reheat large glass substrates that may be prone to stress cracking. For high-speed roll coating systems, abrupt crystal failure can cause great losses in substrates that are not caught in time.

Over the past 30 years, some of the deficiencies of crystal monitors have been addressed and mitigated with crystal, mounting holders and control unit design changes. The use of water-cooled holders, or sensor heads, combined with the use of a marginally temperature insensitive type of quartz (AT-cut), reduced many of the thermally induced frequency shifts for low temperature processes. The use of plano-convex contoured circular quartz crystals mounted in spring contact crystal heads was found to greatly minimize extraneous vibrations. Improved electronics and shielding eliminated much of the radio frequency interference and voltage variations. Subtle corrections to the thickness calculation algorithm have accounted for the acoustic impedance effect. Multiple crystal sensors head, some with as many as 6 crystals available, have been offered as a solution to premature crystal failure.

Nevertheless, film stress, adhesion failure, and extreme temperature effects, including radiation induced frequency shifts, have not been adequately dealt with in sensor design and operation. These have historically been difficult problems to solve and there have not been large enough economic incentives to pursue the solutions. Looking forward to 2003 and beyond, however, the demands of cutting edge research, involving areas such as nanotechnology, biosensors, thin film displays, and high speed optical communications, to name a few, are forcing a re-engineering of the quartz crystal monitor.

### **A Promising Future**

For optical device fabrication, dealing with the issue of thin film stresses as applied to quartz crystals is an area with perhaps the greatest short-term benefit. Up until recently, the solutions for erratic crystal behavior have been best summed up as "art" instead of science. The science is starting to catch up however. Initial systematic studies

of the performance of various crystal designs, including quartz types, electrodes, and crystal geometries has led to some promising new directions for stress tolerant sensors.

The development of the aluminum or aluminum alloy electrode crystal is one such direction. For optical coatings utilizing silicon dioxide, this crystal can extend the useful life of the sensor by 100 to 1000% or more when compared to the industry standard gold crystal. Furthermore, resonance frequency shifts due to electrode adhesion failure are reduced by 90% or more under standard laboratory conditions. This improvement appears to material specific, and in some cases, deposition specific, since it does not eliminate early crystal failure for all dielectrics. It may well prove to be the “test bed” crystal for future thin film stress studies.

The introduction of alternative orientation angles of quartz is a second key area of research for monitor crystals. The standard AT-cut crystal (Figure 2) is obtained by cutting wafers from a bar of cultured, or synthetically produced quartz, at an angle in the vicinity of  $35^{\circ}15'$  from the Z axis. This cut has very little “frequency-temperature dependence” at or near room temperature and has been traditionally used to minimize the resonant frequency shift caused by process heating. It can be varied to operate at higher temperatures as well.

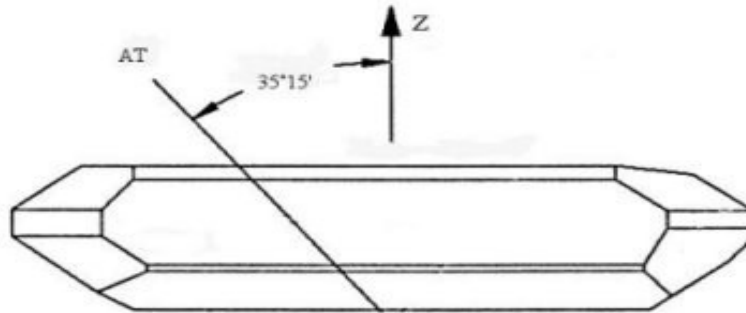


Figure 2: AT-cut from Quartz Bar

A much more promising crystal is based on the SC-cut, or doubly rotated crystal. This crystal orientation exhibits similar “frequency-temperature” behavior of the AT-cut, with the advantages that it shows essentially no “stress-frequency” dependence. A monitor crystal fabricated from SC-cut material, in initial coating trials, exhibits none of the telltale frequency changes induced by high stress dielectrics on AT-cut crystals. Historically this has been a more difficult to produce version of quartz, and hence a higher cost material. The benefits for the optical process engineer may yet outweigh the cost penalty.

The thermal properties of quartz have been given short shrift by the monitor industry since the technology’s inception. An examination of a standard frequency-temperature curve for AT-cut quartz (Figure 4) illustrates how variable the effect of temperature is. If one increases the crystal temperature 20 degrees from room temperature, the frequency will shift approximately 20 Hz. For a material like aluminum, this is equivalent to 10 angstroms of coating. Larger temperature shifts lead to accelerating changes. Given that the temperature swings of quartz during a deposition run

are not monitored in any commercial thin film controller, it should be as no surprise that this hasn't been addressed. Recent studies of standard commercially available sensor heads show that even with water-cooling, the sensor head can rise 20 to 30 degrees within a 10-minute process. For extended runs with high chamber temperatures, this could be considerably larger.

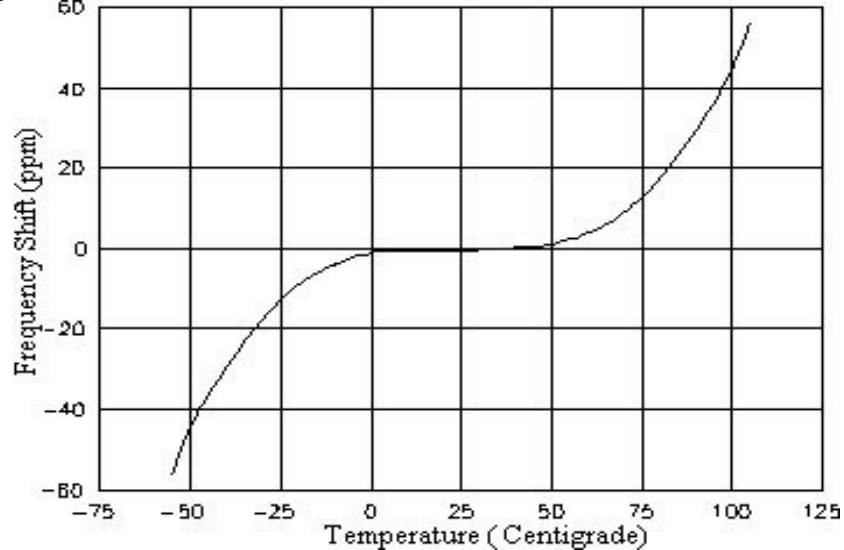


Figure 3: AT-cut Quartz Frequency vs. Temperature Curve

A dedicated quartz crystal sensor head temperature monitoring system that retrofits existing thin film quartz crystal controllers has recently been introduced. With data in hand, the true thermal environment of the crystal sensor can be measured, eliminating the guesswork and adding more science to the optical coatings “art”.

A third direction being taken in the advancement of high reliability quartz monitors, centers on the sensor mounting. Although quartz crystals are capable of sub nanometer measurement and are sensitive to mass accumulations of as little as a picogram ( $10^{-12}$  g), they are traditionally handled by hand by the process operator when installed into the deposition system. In addition to the possibility of cracking or breaking the quartz disk prior to mounting (the sensor is .010” thick and brittle), the contamination of the crystal surface can have disastrous effects on the adhesion of the coating that is being monitored. Adhesion failure results in frequency shifts opposite the magnitude of mass loading, and introduces significant thickness errors and useable crystal life degradation. As a solution to this potential problem, crystals have recently been introduced that are premounted in a holder, with the crystal measurement surface protected from human contact.

A paradigm shift is underway in the understanding of the importance of quartz crystal process monitoring. No matter how significant the breakthrough may be in optical component construction, be it materials, geometry, process design or application, if it requires a thin film coating of any sophistication, the weak link is how accurately that film can be measured. As technology closes in on manipulating Angstrom level properties of matter, the need for reliable thin film metrology rises to a new level of importance. If you can't measure it, you can't control it!