Temperature Measurement and Control of Quartz Crystal Microbalances
Why you should insist on it!

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Quartz crystal film thickness monitors (or QCMs) have been used for years to control vacuum deposition processing of electronic and optical products. The widespread use of QCMs can be attributed to a number of factors: 1) quartz crystals can detect films as thin as one Angstrom, 2) crystals are reliable enough to insure process repeatability, and 3) crystals are relatively inexpensive to implement. When combined with sophisticated electronic control instrumentation, film thickness monitors are extremely easy to use, and give very reliable measurements.

Well, most of the time, anyways!

Anyone familiar with film thickness monitors often comes across a number of problems in their day-to-day use. Most of these problems fall into one of two categories:

A) The measurement given by the monitor doesn't always agree with other measurement methods, especially for very thin films (1000 angstroms or less).
B) The quartz crystal becomes very unstable or abruptly fails during a process run especially with optical coating materials.

In either case, a fancy, state of the art, $10,000 mega monitor with 10 GB of ram quickly becomes a boat anchor!

There are a variety of reasons why these problems are encountered, and fortunately, there are now a variety of solutions available. But first, the basic operation of a crystal monitor has to be examined.

Film thickness measurement is based on the principle that the frequency of vibration of a quartz disc decreases continuously as material is coated on its surface. And, as long as you know the density of the film, a process computer can calculate the thickness versus time. Seems simple enough, right?

Unfortunately this vibration frequency is also sensitive to: 1) temperature changes and 2) stress in the growing film.

Any frequency change in the quartz crystal is interpreted as a film thickness change by the controller or monitor, no matter what causes it! That especially means temperature changes!

To minimize temperature dependence, monitor crystals are cut from a quartz bar at a specific angle called the AT cut. This cut has a near zero frequency-temperature dependence at 20°C. So, for a change of a few degrees in either direction, the quartz crystal will not exhibit any frequency shift. Of course, when you get much past 20 °C, this goes out the window, and the frequency does change, sometimes by great amounts!

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.

OK, what is the temperature of your crystal?

As sophisticated as the electronics have become for quartz crystal thin film measurement, no film thickness monitor manufacturer actually measures temperature. How hard can that be? Instead, they just write in their 300 page manuals that the crystal should be at 20 °C!

In our laboratory, 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 easily experience 20 °C swings during a typical coating cycle. A standard crystal head with 1/8" to 3/16" water lines can not push enough water through to maintain 20° C with hot deposition processes. We had to go to ¼" water lines and high pressure to get anywhere close to tight control!

Without temperature control, you can kiss off your nanotechnology measurements or ultra thin film deposition process control! Just measuring the temperature would allow software correction!

The significance of temperature shifts becomes critical when measuring films less than 500 Angstroms. From 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.

I invented the RC quartz crystal in 2004 to eliminate this radiation-spiking problem. It works very well, but with a temperature controlled or corrected sensor head, it would work a whole lot better.

What really causes early crystal failure? What can I do about it?

Even if you water-cool your crystal sensor to exactly 20 degrees C, you can encounter an even bigger problem. Ever had a crystal abruptly fail when you were coating a substrate with a high stress film such as magnesium fluoride or silicon dioxide? You probably thought it was the dreaded "bad crystal" problem.

Well after making several million crystals, I agree that periodically a few get past QC. But even when a "good crystal" is used, abrupt failure still occurs. In some cases it is simply a spatter caused by the deposition source. That will kill any crystal. But by and large, the greatest source of early crystal failure is stress build-up in the film being monitored.

I minimized this problem when I invented the "alloy" quartz crystal in 1987. This is actually an ultra thick aluminum coated crystal. The extreme thickness of the aluminum minimizes the stress build up in the crystal, leading to significantly longer life, often 100 to 200% longer. It works, and most optical coating laboratories use aluminum instead of the thinner gold electrode crystals. You can make gold thicker, but it tends to dampen out the crystal vibration if you get too extreme.

But the number one way to increase crystal life is to heat the crystal!

In our experiments, we were able to increase the time before failure (or extreme instability) of a gold or alloy coated quartz crystal by heating it to 80 to 90° C. We limited the temperature to 90° C because we wanted to prevent crystal overheating (quartz is not a good material for use over 150- 200° C. For higher temperatures we use gallium phosphate single crystals).

In one series of experiments, we used all the silicon dioxide in a 40 cc electron beam hearth and still the crystals would not fail. We measured crystal life increases of 400 % or more with crystals coated with dielectrics. That means could coat micron level films without failure. What would that be worth in your manufacturing process?

Ignoring crystal temperature, and even worse, not controlling it, leads to the bulk of problems we see in quartz crystal film thickness monitoring. The next generation of film thickness controllers absolutely must have temperature control! The first company that makes one will be truly visionary!