INFICON

APPLICATION NOTE

AUTO–Z[1]: A PATH TO PRECISE CONTROL OF LAYER THICKNESS

INTRODUCTION

Auto-Z, a new method for automatic determination of acoustic impedance ratio (Z-Ratio) of a thin solid film deposited on an AT-cut quartz crystal, can provide significant benefit over the conventional Z-Ratio or Z-factor technique. It is well known that measuring the frequency change of an AT-cut quartz crystal can be used to monitor the deposition rate and thickness of thin solid films.

Sauerbrey [2] first developed the equation for calculating thickness based on frequency change. *Miller and Bolef* [3] improved upon Sauerbrey's equation by incorporating a material's acoustic impedance (Z) into the equation. Later refinement by *Lu and Lewis* [4] resulted in a dramatic improvement to the thickness calculation accuracy. First implemented by INFICON in 1974 as Z-Match®, this quickly became state-of-the-art and remains the fundamental working principle of all quartz crystal microbalance (QCM) instruments. Most current instruments use this fixed correction factor entered in terms of Impedance, Z-Ratio, or Z-Factor. Now further advances in measuring the quartz crystal resonance enable an automatic calculation of Z-Ratio for even greater thickness accuracy.

DRAWBACKS OF CONVENTIONAL Z-RATIO TECHNIQUE

One inconvenience of the conventional technique is that the Z-Ratio of the deposited material must be known. Often the bulk material Z-Ratio is different for a thin film because it is sensitive to process parameters, especially in sputtering and reactive evaporation environments. And for many exotic materials, customized alloys, and sequential layers of two or more materials on the same crystal, this parameter is simply not known. In such cases, the user compromises accuracy by guessing the Z-Ratio (often taking Z = 1.0 or entering the Impedance value as 8.83.) **This can result in a very significant loss of thickness accuracy as is evident from Table 1.**

If a thickness-critical process requires multiple materials to be deposited sequentially, a crystal has to be dedicated for each material. Such is often the case in high-temperature superconductor or optical coatings. Co-deposition of materials to produce alloy films presents similar difficulty. In each case, the user may dedicate a crystal for each material and carefully work out tooling and cross-sensitivity factors for each crystal before starting the process. This is difficult and time consuming, and likely to result in errors.

It is a far simpler task if one has knowledge of an effective Z-Ratio for the material, regardless of its composition. Therefore it seems the need for automatic Z-Ratio determination was always there, for reasons more profound than convenience alone.

INFICON MODELOCK AND AUTO-Z

Only INFICON thin film controllers offer ModeLock frequency measurement. Unlike conventional active oscillator instruments, ModeLock allows measuring of two frequencies, the fundamental and 1st anharmonic. The frequency shifts of these two modes are detected and changes in the crystal's response are used to determine the Z-Ratio. This frequency pair is measured when the crystal is uncoated and the values are stored in the instrument's memory. In subsequent measurements a new frequency pair is detected. Only these two pairs of frequencies, the coating history, and the knowledge of material density is needed. No prior knowledge of a material's Z-Ratio is required.

The complete measurement and information processing cycle can be very short and is performed during each 100 msec interval, 10 times each second. This method is quite general and requires no adjustable parameters. Results of extensive testing on a wide range of materials including metals, dielectrics, co-deposited alloys and sequential layers are presented in Table 1. Excellent agreement is found between the predicted values and the gravimetrically measured mass of deposited films.

In the INFICON IC/5, setting the Z-Ratio parameter in the Sensors screen to "Auto" enables the Auto-Z mode. In the Cygnus instrument, Auto-Z is enabled by setting the Auto-Z parameter in the Process screen to "Yes".

Table 1: Summary of performance comparisons between the Z-Match technique and Auto-Z

Material	Total Thickness (KÅ)	Frequency Shift (MHz)	Bulk Z-Ratio	Automatically Calculated Z-Ratio	Auto-Z Absolute Accuracy Improvement (Reduction)
Aluminum[a]	830	4.0-3.3	1.08	1.10	0%
Ba-Y-Cu[b] layered	229	4.0-3.6	2.1-0.84 -0.44	1.2-1.6	3.8%
Bismuth[b]	83	6.0-5.4	0.79	1.1-1.2	0%
Cadmium[b]	159	6.0-5.0	0.68	0.7-0.88	0.5%
Calcium[b]	249	4.0-3.8	2.62	1.5-1.9	2.6%
Carbon[b]	145	6.0-5.7	3.26	-0.3— -0.5	9.7%
Copper[b]	175	6.0-5.0	0.44	0.5-0.7	(1.7%)
Copper-aluminum[b] layered	249	6.0-5.2	0.44 -1.08	0.7-1.06	2.9%
Cryolite[b]	145	4.0-3.8	1.00	1.0-1.6	1.1%
Ge-Al alloy[c]	150	6.0-5.4	0.52	0.75-0.85	(0.3%)
Indium[b]	132	4.0-3.7	0.84	1.0-1.5	2.7%
In-MgF2[b] layered	189	4.0-3.7	0.84-0.64	1.0-1.7	6.0%
Lead[b]	155	6.0-4.8	1.13	0.93-1.05	3.4%
Manganese oxide[d]	195	4.0-3.5	0.38	1.1-1.2	2.4%
Molybdenum[b]	73	6.0-5.4	0.26	0.6-0.7	1.1%
Nickel[b]	115	6.0-5.3	0.33	0.7	2.6%
Nb-Cu[b] layered	182	4.0-3.5	0.49 -0.44	0.7-1.11	4.7%
Quartz[e]	320	4.0-3.7	1.07	0.7-0.8	(0.8%)
Selenium[b]	139	6.0-5.4	0.86	1.8	6.3%
Si-Al alloy[c]	313	6.0-5.4	0.71	0.9-1.6	0.2%
Stainless steel[a]	255	6.0-4.8	0.35	0.16-0.50	(0.2%)
Tin-indium alloy[b]	126	6.0-5.3	0.84	1.18-1.34	1.0%
Titanium[b]	199	6.0-5.3	0.63	0.9-1.11	(0.7%)
Ti-Al alloy[e]	558	4.0-3.4	1.00	0.38-0.45	3.0%
Titanium nitride[f]	150	6.0-5.3	1.00	0.37-0.43	2.2%

[a] dc sputtering
[b] E-beam
[c] E-beam/codeposition
[d] E-beam/reactive
[e] rf sputtering
[f] dc sputtering/reactive

For a detailed discussion of the theory behind Auto-Z, please see the Technical Paper: *Improving the accuracy of a quartz crystal microbalance with automatic determination of acoustic impedance ratio* by Abdul Wajid, INFICON Inc., published in Rev. Sci. Instrum. 62 (8), 2026-2033, August 1991.

[1] Auto-Z US Patent #5,112,642
[2] G. Z. Sauerbrey, Z. Phys. 155,206 (1959)
[3] J. G. Miller and D. I. Bolef, J. Appl. Phys. 39, 5815 (1968)
[4] C. Lu and O. Lewis. J. Appl. Phys. 43, 4385 (1972)