Next Generation Crystal Microbalances for Thin Film Deposition Monitoring and Control

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The "Achilles heel" of thin film deposition process monitoring and control is the quartz crystal microbalance (QCM). Since its advent in the 1960's, QCM's have been an integral part of most commercial thin film coating systems. Unfortunately, the limitations of QCM's for processes such as optical coating, ion beam sputtering, MBE and CVD have not been adequately addressed.
A new class of QCM sensor, designed for elevated temperatures, high stress dielectrics and extremely thin coatings is now available. This new sensor embodies an advanced crystallographic cut, "smart" sensor housings with integrated heaters, and novel crystal materials to extend the range of thin film monitoring to up to 250 C.

INTRODUCTION

Quartz crystal microbalances operate in a relatively simple fashion. The QCM consists of a disc of quartz cut at a specific angle and shape from a bar of synthetically grown quartz. This quartz disc is then coupled into an electrical circuit and caused to vibrate at its natural resonance frequency. The resonance changes (decreases in frequency) whenever a thin film coating collects on the crystal surface. If the density of the film material is known, an algorithm can be used to compute the film thickness.

PERFORMANCE LIMITATIONS

The use of QCM’s for thickness monitoring and rate control is not without difficulties. Users find that quartz crystals are ideal for the deposition of thin metal films since they cause a reproducible, stable change in the crystal resonance frequency. But when coatings of dielectric materials such as silicon dioxide or magnesium fluoride are monitored, the crystal can become very erratic or even abruptly fail. This is the result of strong tension or compression generated in these films. As quartz is stress sensitive, the QCM responds to these forces and often becomes unstable.

The heat of optical thin film depositions also adversely affects QCM’s. Quartz is heat sensitive and must be water-cooled in order to minimize any changes of resonance frequency with temperature. But even with cooling, the QCM will respond to radiant heat from a deposition source and will be prone to large thickness errors when monitoring very thin films (<100 A).

Finally, the temperature limitation of quartz has severely limited its application in coating technology. The practical upper limit of quartz resonators is approximately 150ºC, well below the temperature required for many thermal and chemical vapor depositions. As quartz is heated, it undergoes a change in frequency, the slope of which gets steeper the higher the temperature goes. This steep slope makes for very difficult frequency measurements, since a small rise in temperature leads to a large change in resonant frequency.

PERFORMANCE IMPROVEMENTS

The shortcomings of QCM’s have been addressed by three new technologies: 1) an advanced crystallographic cut of quartz, called the RC™ or radiation compensated crystal, insensitive to radiant heat from deposition sources, and as a result, extremely accurate for nanolayer thin film depositions; 2) temperature regulated QCM sensor housings that precisely control the crystal temperature during depositions and, by operating at an elevated temperature, 250° C and higher, minimizes thin film stress and extends crystal life dramatically, and 3) new piezoelectric materials that are quartz structural homologues, giving all the piezoelectric qualities of quartz but with a higher range of operation (900ºC and up). These crystals pave the way for real-time CVD sensors and a variety of research applications.

FUTURE DEVELOPMENTS

The advancement of the QCM will be further enhanced by future products in development: 1) Multi-resonator quartz crystals consisting of several discrete vibrating areas on a single piece of quartz, offering redundant, “failure proof” crystal sensors, 2) QCM’s which incorporate thin film stress measurement functions into the sensor body, allowing real-time monitoring of compression and tension in the coating as it deposits, 3) Pre-packaged crystal sensors, eliminating the need for handling the QCM and mounting it in the sensor housing (a major source of crystal problems) and 4) Wafer sliced, miniature crystal sensors, which will drastically reduce the price of QCM’s used in thin film monitoring.