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**QCM Breaking News!** 

The next generation Crystal Microbalances for Thin Film Deposition monitoring and control have arrived!

# **Thin Film Solutions**

### A Problem Solver Guide to PVD & CVD Process

ALD / CIGS / OLED / Optical Coating / CVD Solar Cells

# The Next Generation Crystal Microbalances for Thin Film Deposition Monitoring & Control have arrived!

We're positioned to strongly impact the market with innovative film thickness measurement sensors, hardware, and electronics for thin film applications such as **OLED**, **CIGS**, **ALD** & **optics**.

Many things exciting have been happening in the field of deposition measurement in 2012:

- In October 2012 we shipped to a major European electronics manufacturer (household name) their first of the new generation TEMPE<sup>™</sup> heated sensor holder and EON<sup>™</sup> Thin Film Instrument, a new generation compact deposition controller.
- Atomic Layer Deposition (ALD) sensor in an important thin films lab of a major research institute in the eastern part of Germany, for testing on a 300mm silicon wafer line.
- ▲ A prestigious OLED research institute in Germany to install a TEMPE<sup>TM</sup> OLED self-cleaning film thickness sensor in their prototype facility.
- Patent application filed for the world's first high flux continuous run film thickness monitor sensor. In simpler words, now you can measure zillions of angstroms of thin film coatings real time without failure. Never been done before with Angstrom level accuracy! This will be a boon for OLED and metallized plastic film (as for food packaging) and thin film solar cell manufacture (e.g. selenium deposition).
- Demonstrated in lab environment the first production-ready film thickness sensor (microbalance) with continuous operation at 600°C. That's a world record for a commercially available film thickness system (the Tempe<sup>™</sup> sensor head and Eon<sup>™</sup> controller). This work was supported by the US Department of Energy. Also, in a research mode only, demonstrated over 700°C operation, hot enough to re-evaporate Indium and Gallium off the surface of a Nickel electroded crystal.



Eon™ Deposition Controller



Tempe<sup>™</sup> self-cleaning sensor



Phoenix™ Sensor



EonLT<sup>™</sup> Thin Film Monitor



Helios<sup>™</sup> sealed sensor head

### **Atomic Layer Deposition**

#### PROBLEM

- Traditional quartz crystal microbalances (QCM's) do not work in ALD systems because the crystal surface must be at the decomposition temperature of the gaseous precursors
- Traditional QCM's do not work in ALD systems because the sensor head does not have temperature measurement or control

#### SOLUTION

- Heated sensors with integral temperature measurement and specially constructed high temperature quartz crystals can be operated up to 500°C, duplicating ALD conditions identical to the substrates being coated
- Waterless unheated sensors that can withstand up to 500°C
- Temperature controlling and monitoring instrumentation

#### **RECOMMENDED PRODUCT**

- Colnatec RC<sup>™</sup> Quartz crystals (up to 250°C)
- Colnatec SuperQuartz<sup>™</sup> (up to 1000°C)
- Tempe<sup>™</sup> heated 100°C 500°C Sensor Head
- Eon<sup>™</sup> Deposition Controller



#### PROBLEM

Low temperature ALD (<150°C) cannot be accomplished with quartz due to high noise</li>

#### SOLUTION

- Patented, noise resistant crystals that can be operated at 200°C with or without an integral heater in the sensor head, allowing real-time ALD measurement
- Temperature compensating crystal instrumentation eliminate rate noise

- Colnatec RC<sup>™</sup> quartz crystals or SuperQuartz
- Tempe<sup>™</sup> Sensor Head
- Eon<sup>™</sup> Deposition Controller

### CIGS - Copper Indium Gallium Selenium process for flexible solar cell production

#### PROBLEM

 Inline coating systems require long run times to create economical solar cells; the run times cause excessive crystal loading leading to failure

#### SOLUTION

Self-regenerating crystal systems provide extended run times, allow full process runs

#### **RECOMMENDED PRODUCTS**

- Helios<sup>™</sup> self-cleaning, sealed sensor head for selenium depositions
- Tempe<sup>™</sup> self-cleaning sensor head for materials other than selenium
- Eon<sup>™</sup> temperature controlling deposition controller and (▲T) noise correction
- Colnatec SuperQuartz<sup>™</sup> or RC<sup>™</sup> quartz crystals



#### PROBLEM

- High Temperature deposition conditions cause rate noise and crystal failure due to the low temperature limitations of quartz
- Layers such as Selenium cause high noise due to high dissipation factor

#### SOLUTION

- Specially constructed high temperature sensors can operate at temperatures in excess of 500C with very stable frequency and rate behavior
- Patented, noise resistant quartz crystals and temperature compensating instrumentation

- SuperQuartz<sup>™</sup> or HT (high temperature) quartz crystals
- Tempe<sup>™</sup> or Helios<sup>™</sup> sensor head
- Eon™ Deposition Controller for high temperature control and (▲T) noise correction

### CIGS (cont.)

#### PROBLEM

• Heavy buildup of materials, such as selenium, cause crystal failure due to loss of crystal activity

#### **SOLUTION**

- Heated crystals increase the crystal lifetime by reducing film stress and increasing crystal activity ("Q")
- Temperature controlled, self-cleaning sensor heads and instrumentation for regenerating crystals to "like-new" condition

- Tempe<sup>™</sup> self-cleaning sensor head or Helios<sup>™</sup> sensor head
- Eon<sup>™</sup> Deposition Controller





### **OLED: Organic Light Emitting Diode**

#### PROBLEM

- Long deposition run times, particularly with in-line systems, are not possible due to limited crystal life
- Crystals fail at or around 10 hours

#### SOLUTION

- Elevated crystal sensor operation temperatures, up to 90°C, minimize crystal noise and extend crystal life
- Temperature controlled sensor heads and instrumentation

#### **RECOMMENDED PRODUCTS**

- Tempe<sup>™</sup> self-cleaning sensor head
- Eon<sup>™</sup> Deposition Controller
- Colnatec RC<sup>™</sup> or High Temperature (HT<sup>™</sup>) crystals



#### PROBLEM

- Noisy crystal behavior (rate noise +/- 50% of set point signal) leads to inaccurate thickness measurement and early crystal failure
- Deposition rate is difficult to measure because the crystal reading has excessive rate noise

#### SOLUTION

- The superior finish AT-cut X-TRONIX Quality Crystals are designed to ensure low noise operation due to special surface treatment
- Specialty cut crystals reduce noise as a result of their insensitivity to stress and radiant heat
- Temperature controlled sensor heads and instrumentation

- The superior finish AT-cut X-TRONIX Quality Quartz Crystals, or Colnatec RC<sup>™</sup> or High Temperature (HT<sup>™</sup>) crystals
- Tempe<sup>™</sup> self-cleaning sensor head
- Eon<sup>™</sup> Deposition Controller

### OLED (cont.)

#### PROBLEM

- Measurement of thin (<100 Angstrom) layers is error prone due to thermal shock of the sensor crystal upon exposure to the deposition source
- When the shutter on the deposition source is opened, the crystal rate spikes then settles, causing the rate reading to be obscured

#### **SOLUTION**

 Colnatec RC<sup>™</sup> and High Temperature (HT<sup>™</sup>) crystals are impervious to thermal shock and result in high accuracy

- Colnatec RC<sup>™</sup> or High Temperature (HT<sup>™</sup>) crystals
- Tempe<sup>™</sup> self-cleaning sensor head
- Eon<sup>™</sup> Deposition Controller



### **OPTICAL COATING**

#### PROBLEM

 Optical thin films such as MgF<sub>2</sub> and SiO<sub>2</sub> cause crystals to become excessively noisy and/or fail early, preventing completion of deposition run

#### SOLUTION

- Heating crystals to 90°C allows extended crystal life by reducing stress, thereby eliminating noise and crystal failure, often by as much as 500%
- Alloy quartz crystals significantly reduce film stress leading to low noise and long crystal life
- Temperature controlling instrumentation

#### **RECOMMENDED PRODUCTS**

- Tempe<sup>™</sup> self-cleaning sensor head + Eon<sup>™</sup> Deposition Controller
- Phoenix<sup>™</sup> Sensor + EonLT<sup>™</sup> Thin Film Monitor
- Alloy quartz crystals (standard or premium AT or the stress-resistant RC)



#### PROBLEM

 Multilayer dielectrics consisting of 50 or more layers exceed the capacity of crystals due to excessive film buildup

#### SOLUTION

- Heated sensor heads increase crystal life by 500% or more
- Temperature controlling instrumentation

- Tempe<sup>™</sup> self-cleaning sensor head + Eon<sup>™</sup> Deposition Controller
- Phoenix<sup>™</sup> Sensor + EonLT<sup>™</sup> Thin Film Monitor

### **OPTICAL COATING** (cont.)

#### PROBLEM

- Ultra-thin film layers used in "needle" synthesis optical film manufacture are difficult to measure due to crystal rate shock upon opening of the source shutter
- When the shutter on the deposition source is opened, the crystal rate spikes then settles, causing the rate reading to be obscured

#### SOLUTION

- Noise resistant crystals are impervious to thermal shock (minimizing stress) and result in high accuracy
- Temperature controlling instrumentation

#### **RECOMMENDED PRODUCTS**

- RC<sup>TM</sup> quartz crystals
- Phoenix<sup>TM</sup> Sensor + EonLT<sup>TM</sup> Thin Film Monitor



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### In the Pipeline: CVD for Solar Cell Manufacturing

The production of polysilicon for solar cells is based on the TC3 or Siemens process wherein a gaseous silicon compound, trichlorosilane (SiHCl3 or TC3) and hydrogen are fed into a process chamber where it makes contact with silicon rods heated to near 1100°C. The TC3 pyrolyzes or breaks apart into a thin layer of silicon and gaseous hydrogen chloride, the latter being pumped away as a waste product.

As pioneers in high temperature microbalances, or mass measuring devices, Colnatec is developing a 1000°C sensor that can duplicate the hot silicon rod surface. This sensor can, in real time, measure the deposition rate of the polysilicon film as it pyrolyzes. Such information can be used to monitor, control and even fine tune the chemical kinetics of the polysilicon process, resulting in increased yield, lower energy costs and even higher throughput. The sensor can also be used to monitor the composition of the incoming and exhaust streams using a tunable temperature controlled housing.

Additional applications for this revolutionary sensor include controlling the kinetics of the TC4 to TC3 reconversion process, detection of waste products in process scrubbers, and basic research investigations into high temperature CVD and gas phase reactions encountered routinely in silicon solar cell production systems.



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### The Current Family.... and growing fast!



Eon<sup>™</sup> Deposition Controller



Tempe<sup>™</sup> self-cleaning sensor



Phoenix<sup>™</sup> Sensor



EonLT<sup>™</sup> Thin Film Monitor



Helios<sup>™</sup> sensor head



- Eon<sup>TM</sup> Thin Film Deposition Controller
- EonLT<sup>™</sup>, World's Most Economical Thin Film Thickness Monitor
- Helios<sup>™</sup> Hermetically Sealed, Crystal Sensor (to 800°C)
- Tempe<sup>™</sup> Sensor Head: Patented High Vacuum Crystal Sensor System (to 500°C)
- AT-cut Quartz Crystals: Standard Replacement Quartz Crystals for Film Thickness Monitoring
- RC<sup>™</sup> Quartz Crystals: Superior Replacement Quartz Crystals for Precision Film Thickness Monitoring
- HT<sup>TM</sup> Crystals: High Temperature Crystals for Self-Cleaning & Sealed Sensors
- SuperQuartz<sup>™</sup>: Superior Crystals for Harsh Process Film Thickness Monitoring
- NB: Development continues on the high temperature front for sensors and higher values have been reached under laboratory conditions.

# Patented RC<sup>™</sup> Crystal Proven Most Accurate Film Thickness Sensor

Independent German test lab confirms RC<sup>™</sup> crystal non-responsive to thermal shock.

December 2, 2011 - Colnatec LLC, thin film industry's leading designer and manufacturer of high-end sensors, control systems and quartz crystals for deposition systems, has released the news that their patented RC<sup>™</sup> quartz crystal has been independently tested and verified by a German thin film lab to be thermal shock resistant. Thermal shock is responsible for thickness errors that can destroy today's most sensitive electronics, so a shock-resistant crystal enables manufacturers to reduce yield loss and improve film thickness control.

"The RC<sup>™</sup> represents the solution to a long-standing film thickness measurement problem--the spiking of the rate when a crystal is first exposed to source radiation," said Colnatec CTO, Scott Grimshaw. "The RC<sup>™</sup> crystal gives our customers, particularly thin film lighting and display manufacturers, a significant edge in controlling their process."

The measurement of film thickness during a vacuum deposition process can be accomplished with great accuracy and precision using a quartz crystal microbalance, or QCM. A film thickness sensor measures the change in resonance frequency of an oscillating quartz crystal while a thin film coating is collecting on its surface. Under controlled conditions, it is possible to achieve atomic level resolution of the film thickness. In practice, however, this is rarely achieved.

As the coating builds up on the surface of the crystal, the resonance frequency decreases. For ultra-thin film thickness measurements, such as in Organic Light Emitting Diode (OLED) displays or solar cells, a combination of frequency shifts caused by thermal shock, stress and film build-up can lead to thickness errors of 100% or more. As a result, the efficiency and functionality of the electronic device is seriously compromised.

Because the RC<sup>™</sup> crystal will not show a rate spike when the deposition source shutter is opened, and it maintains a consistent reading as coating accumulates, thickness measurements are more accurate. Sub-nanometer thin films are almost impossible to measure with standard crystals because the thickness overshoots during the thermal shock period.

"It is quite impressive that the RC quartz doesn't show any negative effect on the shutter action," stated Ralph Kempter, founder and lab test director at BeamTec, Blaustein -Germany. "Also there's no drift when the shutter is closed. Really impressive."

RC<sup>™</sup> crystals can be used in place of standard AT-cut quartz in all commercially available film thickness monitors and controllers. They provide significant advantages in the measurement of nanometer films used in the manufacture of OLED's, precision optical interference films, or next generation electronic devices.





Patented Quartz Crystal - non-responsive to thermal shock, most accurate QCM sensor in the world!

Traditional Quartz Crystal - used on most QCM deposition controllers on the market

