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Development of a real-time transient cycle mass monitor

Quartz Crystal Microbalance – D R Booker

Introduction

The most common dynamic instruments for particle mass monitoring have been the Beta Attenuation, and Tapered Element mass monitors (e.g. TEOM). Quartz crystal microbalances were popular in the 1970's and 80's, but because the sensor is easily overloaded with mass the TEOM and Beta devices became dominant for ambient PM_{10} dust monitoring. However, with the current emphasis on ultrafine particles in the environment (e.g. those emitted from gasoline and diesel vehicles) in which the mass concentrations are typically much lower, and the drive is to make them lower still, the greater sensitivity of the quartz sensors makes this technique more suitable to modern applications. Since the 1980's a great deal of development work has been carried out to measure very low masses.

Quartz Crystal Microbalance

The Booker Systems' *QCM* devices dynamically determine the amount of aerosol particles deposited on a substrate from a known volume of air. A piezoelectric crystal (see picture above) is used as a sensitive microbalance. Electrostatic precipitation collects aerosol particles on the surface of the piezoelectric crystal. The crystal is excited in its natural frequency, which decreases with increasing mass load on its surface. Thus, the particulate mass collected on the crystal can be determined by measuring the change in the crystal's natural frequency. This is an ideal solution for measuring environmental and ultrafine particulates. As large particles (around 5µm and above) cannot couple to the crystal, they do not contribute to the measured mass of a microbalance. In addition, the QCM has been designed with a $PM_{2.5}$ inlet. To ensure that particles are attracted and couple to the crystal surface a point-to-plane electrostatic precipitator is used. The collected mass on the crystal can be retained for further analysis or the crystal can be cleaned and replaced.

Software

The *QCM* can be controlled by using supplied software based on a *Labview®*[2] platform. The software allows the instrument to be configured against specific measurement requirements as well as providing graphical presentation of data. The recorded files can be exported to a range of spreadsheet programs. In addition the *QCM* can be used to accept data from other measurement or sample conditioning devices through its CAN interface so that the data can be collected into a single file for subsequent analysis.

Integration

By using the CAN interface, the *QCM* can report to or receive reports from a range of products. If a number of measurement devices produce analogue or serial outputs then the Booker Systems *Datalogger* can combine these output streams into a single report. The *Sample Conditioning System* will present a concentrated sample to the *QCM* in an accurately diluted form.

Outline specification

Microbalance	0.465 Hz/ng resolution gold or
Crystal surface	platinum
Power	85-240V AC ~50Hz
Communications	CAN and 9 pin Serial
	GPS Port
Dimensions	Analogue input
Software	D322 x W255 x H160mm
	Labview® V.4 Application[3]

[2] *Labview is a registered trademark of National Instruments Ltd.*

[3] *This software requires a minimum Pentium PC with VGA graphics.*

Quartz Crystals

Introduction

The *Quartz Crystal Microbalance* (*QCM*) sensor is used to measure the particle concentration of an aerosol at a range of temperatures. Within the instrument, a piezoelectric crystal is exposed to a stream of electrostatically-charged particles that deposit on the surface of the crystal. By measuring the frequency-shift of the crystal and knowing the mass sensitivity (frequency to mass-added factor for that crystal), the mass accumulated can be determined. Provided that the volumetric gas flow is also measured, a mass concentration can be determined.

Crystal Frequency

The piezoelectric quartz crystal is externally driven by an electronic oscillator attached to two metal plates (usually deposited by vacuum evaporation) placed on either side of a quartz blank.

This imposes a time-dependent electric field across the plate, which causes the crystal to oscillate at a frequency determined by the total thickness of the crystal plus any mass on the outsides of these electrodes. The oscillation appears as a Gaussian distribution of displacement, peaking at the centre and vanishing at the electrode edge. The frequency of the surface motion decreases as a layer of contaminant is formed (mass addition), according to the degree to which each element is being displaced by the oscillation. The arriving or departing molecules (mass flux) are deposited or desorbed randomly. Therefore, integrating the distribution of surface displacements provides a valid sensitivity (mass flux to change in frequency) for the quartz plate. Experiments have proved that the mass sensitivity of the quartz crystal is as predicted.

The resonant frequency of the *QCM* used is normally 5MHz, but this can be varied depending on the application. The piezoelectric quartz crystal is approximately 14mm in diameter. Normally, the quartz plate electrodes have different diameters on the two surfaces. Electrodes of gold, platinum, and other metals can be supplied. The electrode- to-crystal outer diameter ratio is usually approximately one half, in order for the crystal to have a high "Q" (activity coefficient). While one of the electrodes must have this ratio to contain the electric field, the other side of the quartz crystal may have an electrode that covers the blank completely (see Crystal Figure). The controlling electrode is the one smallest in diameter. (The smaller of the two electrodes defines the "active area" of the crystal.) Normally, this controlling electrode diameter is 6.4mm, which results in an active area of 0.32 cm^2. Molecules that strike the crystal outside the active area do not affect the crystal frequency, even though the crystal is wholly plated. The completely coated surface is normally exposed to the particles. The “actual” active area in which the particles are deposited is smaller than the “electrode active area” in the *QCM* because of the design of the electrostatic precipitator which promotes deposition onto the crystal.

Mass Sensor Range

The usual stated mass sensor dynamic range is 1/100 of the resonant fundamental frequency, i.e. 50,000 Hz for a 5 MHz crystal. This means that the sensitivity can be assumed to be essentially constant over that range. This is true only if the deposit is a solid polycrystalline or amorphous layer. If the deposit consists of liquid or solid particles, then this range may be considerably reduced due to damping of the crystal oscillation. For $PM_{2.5}$ aerosol applications, the sensor dynamic range is 1/1000 of the resonant fundamental frequency, i.e. 5,000 Hz for a 5 MHz crystal. The overloading capacity is much lower than for a uniform layer over the entire active area because in the *Booker Systems* QCM the deposit area is controlled by the electrostatic precipitator design, and is typically much smaller than the active area governed by the electrodes.

Sense and Reference Crystals

A crystal's frequency is sensitive to temperature and mass. A curve describes the frequency versus temperature variation.

The reference and the sensor crystals are usually selected so that the beat frequency of the clean two-crystal assembly at the desired "centre" temperature is in the range of 2-5 KHz to avoid any "lock-on" of two oscillators. The sensor crystal assembly has the lower frequency with respect to the reference crystal, since added mass on the crystal *lowers* the frequency of oscillation. Otherwise, the beat frequency will not be a monotonic function of mass. Matching the frequency of the sensor and reference crystals, while "clean" of contamination, allows the same comparison at that temperature when the crystal becomes "contaminated" with deposited mass.

In practice, the beat frequency versus temperature for the clean condition is never ideally matched, although a simple polynomial equation can usually be used to describe the *QCM*'s behaviour if it is repeatable. A thoroughly repeatable test in a "contamination-free" environment will assure frequency repeatability with temperature, even with hysteresis effects, i.e., when decreasing temperature does not give the same frequency as increasing temperature. This hysteresis results, at least in part, when the sense and reference crystals are not isothermal. Obviously, good thermal contact between the crystals and temperature sensor is important if the sensor is to portray the actual temperature of the crystals.

Mass Sensitivity of Crystals

Crystal Temp.	Mass Sensitivity	f = 5 MHz	f = 10 MHz
25°C	S = Hz/g/cm^2	5.6569 x 10^7	2.2627 x 10^8
	1/S = g/cm^2 -- Hz	1.7677 x 10^{-8}	4.4195 x 10^{-9}