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SURVEY ON MASS DETERMINATION WITH OSCILLATING SYSTEMS Part II. Instruments and weighing of matter from gaseous environment

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Abstract

Examples of available instruments based on oscillating systems are discussed including quartz crystals, vibrating strings, ribbons and other bodies.

Keywords: balance, oscillation, quartz, tapered element, vibrating string

Single crystal balances

A single crystal microbalance (CM) employs a sensor that consists of a thin crystalline disc sandwiched between two electrodes. Small oscillations in the sensor at resonance frequencies are created by applying an AC voltage across the electrodes. Extremely high resolution up to 1 pg can be achieved with CMs and a relative sensitivity up to 10^{-6} is attainable. Piezoelectric crystals combine long-term stable behaviour with little damping, good constancy of the elastic properties as well as low sorption. A CM is distinguished by its simple design, small size and low cost. The measuring equipment is robust, resistant to aggressive surroundings, shock-proof and not sensitive to ground vibrations. Its output quantity, the frequency, is easily and fast registered and can be directly digital processed. The balance creates its own acceleration field and is independent of gravitation. For mass determination with a CM, the following fundamental conditions must be fulfilled [1]:

- The mass must consist either of a homogeneous film that evenly covers the oscillating region of the single crystal, or of evenly distributed fine particles.
- The mass must completely adhere rigidly to the quartz surface.

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1418–2874/2003/ \$ 20.00 © 2003 Akadémiai Kiadó, Budapest Akadémiai Kiadó, Budapest Kluwer Academic Publishers, Dordrecht The resonance behaviour of a piezoelectric single crystal is described by an electrical equivalent circuit diagram consisting of the parallel connection of an acceptor circuit with a capacitor (Fig. 1) [2]. This system has a parallel and a serial resonance. Only an operation with serial resonance corresponding to the mechanical resonance frequency can be used in weighing.

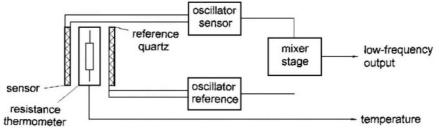


Fig. 1 Diagram of a quartz measuring system

As a rule, a single crystal resonator produces only one output quantity, and that is the change in resonance frequency. If several measurement quantities are to be registered several crystals are to be used. Another method makes use of the different sensitivities of resonance frequencies of different modes in one crystal. This method is applied, for instance, in simultaneous mass and temperature determinations. In order to eliminate temperature or other disturbing influences comparison with the signal of a reference crystal is recommended (Fig. 2).

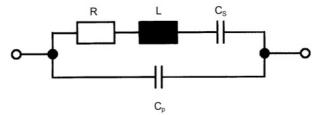


Fig. 2 Equivalent circuit of a single crystal resonator

Today mostly used is the quartz crystal. Following examples give some idea to the variety of both sensor development and application of the quartz crystal microbalance (QCM).

Oscillators are ideal from the point of view of vacuum technology because of their small surface and their bakeability, and they are not affected by buoyancy. For quite some time now the quartz crystal microbalance has served to control and regulate thin-film deposition processes, as in vacuum metallising, sputtering and ion implantation. Thickness of deposited layers can be monitored in situ. The QCM is a reliable instrument for the automation of these processes, a condition for reproducible results and consistently high quality. Furthermore QCMs are used in the quantitative determination of surface contamination.

For measurements in gravity-free space, mass must be determined either indirectly, or weighing systems must be used that generate their own acceleration field as

is with oscillation systems. One of the typical tasks is the monitoring of the technical functions of objects, surface contamination and evaporation in space (Fig. 3), and those in simulation chambers.

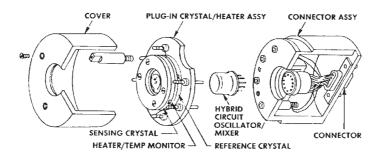


Fig. 3 Berkeley quartz crystal microbalance designed for contamination records in space

Another area of application is testing of new materials with regard to evaporating components. A device for determining outgassing rates has been described by Glassford [3]. It contains an effusion cell whose molecular mass flow escapes into the space around the quartz balance through an opening equipped with a sealing cap.

Recently quartz-homeotypic gallium orthophosphate $GaPO_4$ is recommended as crystal material [4]. It is suitable for operation up to 970°C. Plano-convex resonators of Y-cuts with 10 mm diameter have been used as sensor for that CM whereby high temperature stability was achieved by means of compensation.

Vibrating string

To increase the frequency of a note produced by stimulation of a string in a musical instrument we increase its tension. Likewise, by increase of the mass suspended on a rope or chain the frequency of a standing wave increases. Because for loads of several kilograms these frequencies are low, it can easily be observed and a sensitivity of about 1 percent may be attained. This method is applied e.g. when hoisting sunken ships.

Load cells equipped with vibrating string sensors are applied in commercial scales as well as in belt weigher.

Vibrating ribbon

Considerations that an oscillating substrate of lower quality than quartz should be suitable for many applications, if the ratio of precipitated to substrate mass is large enough, leaded to development of longitudinally and transversally vibrating ribbon, as it provides measurement and documentation feasibility, e.g. of the mass rate *vs.* time. Pertinent experiences comprise experiments in vacuo [5], dust measurements (Fig. 4) [6, 7] and the measurement of solid particles in water [8] with the transversally oscillating ribbon and dust concentration, viscosity and elasticity measurements with the longitudinally vibrat-

ing ribbon [9, 10]. The strong dependence of the eigenfrequency on temperature and mechanical exertions required among other things a sophisticated thermostat. Therefore considerations to improve the measurement method were made by modelling the object patch more strictly and getting the results by calculating the quotient of sensitive to non-sensitive eigenvalues. Further quantities may be detect by measuring more than two eigenfrequencies [11].

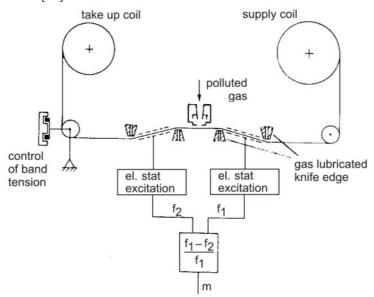


Fig. 4 Differential system for dust measurements with the aid of a vibrating band

Vibrating body

Tuning fork shaped bodies stimulated to oscillations were applied as sensors for laboratory balances.

For routine particle deposition a tapered element oscillating microbalance (TEOM) was developed. TEOM consists of a thin-walled tapered element, its broad end fixed to a solid base. The free end executes autonomous oscillations, the frequency of which depends on the mass and the stiffness of the element and on the additional mass connected to the free end. The oscillations are excited electrostatically, and detection is optoelectronic. The instrument is calibrated by connecting it to a reference mass. From the differences between the oscillation frequencies with and without the reference mass, a constant is determined which corresponds to the spring rate of a discrete mechanical oscillator [12]. The measuring range is between ten to several hundred mg. Areas of application are the continuous measurement of particle emissions of diesel machines (Fig. 5) [13] and monitoring of mass changes by deposited dust particles in atmosphere control.

A vibrating oval metal tube of thin sheet metal, closed at one end, was used as difference pressure sensor in the range of high vacuum to several bars.

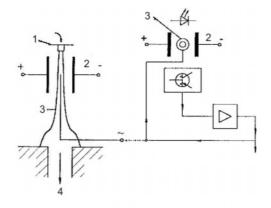


Fig. 5 Tapered element oscillating microbalance. 1 – filter, 2 – electrodes, 3 – conus shaped tube, 4 – to pump. The contaminated gas is pumped through a filter at top of the conus shaped tube and the mass of dust deposited is determined by shift of frequency of the tube in the field of two electrodes

By use of carbon nanotubes of diameter up to 75 nm and length of a few micrometer, which were resonantly excited in the flexural mode by an alternating voltage of a few MHz, the mass of attached spheroidal carbon particles has been determined to 22 ± 4 fg [14].

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