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Researchers link quartz microbalance measurements to international measurement system

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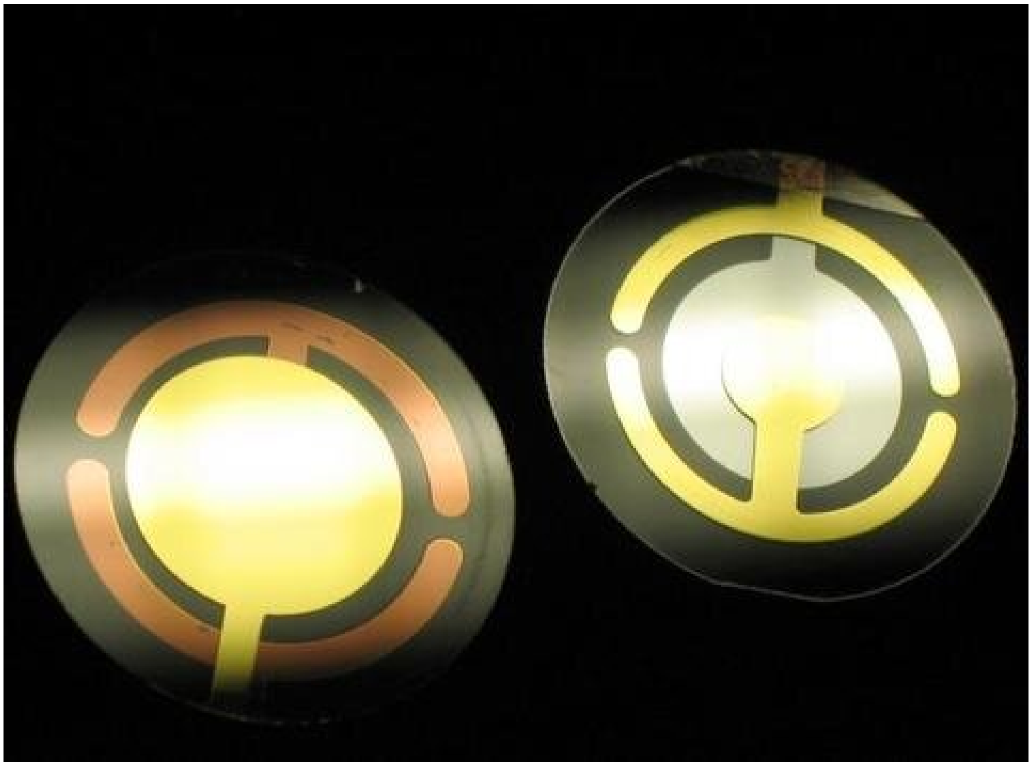

Opposite sides of a quartz crystal resonator. Credit: Beaker via Wikipedia

Researchers at the National Institute of Standards and Technology (NIST) have found a way to link measurements made by a device integral to microchip fabrication and other industries directly to the recently redefined International System of Units (SI, the modern metric system). That traceability can greatly increase users' confidence in their measurements because the SI is now based entirely on fundamental constants of nature.

The device, a dime-size disk called a quartz crystal microbalance (QCM), is critically important to businesses that rely on precision control of the formation of thin films. Very thin: They range from micrometers (millionths of a meter) to a few tens of nanometers (billionths of a meter, or about 10,000 times thinner than a human hair) and are typically produced in a vacuum chamber by exposing a target surface to a meticulously regulated amount of chemical vapor that sticks to the surface and forms the film. The greater the exposure, the thicker the film.

Thin films are essential components in electronic semiconductor devices, optical coatings for lenses, LEDs, solar cells, magnetic recording media for computing, and many other technologies. They are also employed in technologies that measure the concentration of microbial contaminants in air, pathogens in the water supply, and the number of microorganisms that attach themselves to biological surfaces in the course of infection.

All those uses demand extremely accurate measurements of the film's thickness. Because that is difficult to measure directly, manufacturers frequently use QCMs, which have a valuable property: When an alternating current is applied to them, they vibrate at a resonant frequency unique to each disk and its mass.

To determine exactly how much film material is being deposited, they place the a QCM disk in the vacuum chamber and measure its resonant frequency. Then the disk is exposed to a chemical vapor. The more vapor that adheres to the QCM, the greater its mass—and the slower it vibrates. That change in frequency is a sensitive measure of the added mass.

"But despite ubiquitous implementation of QCMs throughout industry and academia," said NIST physicist and lead researcher Corey Stambaugh, "a direct link to the SI unit of mass has not existed." The relationship between the SI unit of mass (the kilogram) and resonance frequency is assumed to be well characterized after decades of QCM measurements. But over the years, industry has made inquiries to NIST regarding the absolute mass accuracy of these frequency measurements. The new results presented by Stambaugh and colleagues are in large measure a response to those queries.

"We expect that our findings will enable a new, higher level of assurance in QCM measurements by providing traceability to the new SI," said NIST physicist Joshua Pomeroy, who with Stambaugh and others report their findings today in the journal *Metrologia*. The redefinition of the SI units in May 2019 eliminated the previous metal prototype kilogram as a standard and instead defined the kilogram in terms of a quantum constant.

In the new SI, mass at the kilogram level will be realized in the United States using that constant in NIST's Kibble balance.

In the new SI, NIST They have also developed a standard instrument, called the electrostatic force balance (EFB), that provides extremely accurate measurement of masses in the milligram range and lower), which are directly linked to the SI by way of a quantum constant. The EFB provided the team with reference milligram sized-masses with a precision on the order of a fraction of a microgram (1/1,000,000th of 1 gram, or about one millionth the mass of an average paper clip).

Stambaugh and colleagues carefully weighed an uncoated quartz disk, then suspended it in a vacuum chamber and measured its resonant frequency. About 0.5 meters (20 inches) below the disk was a furnace that heated a quantity of gold to 1480 C (2700 F). Gold vapor from the furnace rose and attached itself to the lower surface of the QCM, increasing its mass and thus slowing its resonant frequency. The scientists repeated the procedure at different time intervals and thus different amounts of mass accretion. was repeated at different time intervals. The researchers deposited gold vapor was over different time intervals and recorded the subsequent changes in resonant frequency. They weighed the disk again using the same EFB reference masses. This provided an accurate measurement of the change in mass, and thus provided an exact measure of the amount of gold deposited.

In the course of the work, the team also performed a complete assessment of the uncertainties in the QCM measurements. They identified the most accurate mathematical method of correlating the addition of mass to the change in the QCM's resonant frequency.

"This work provides a key step in a technique for traceably tracking—and thus correcting for—mass changes over time," said NIST physicist Zeina Kubarych.

In that regard, the new findings could help improve the way mass is disseminated following the new SI definition. The new kilogram is "realized"—converted from an abstract definition to a physical reality—through highly controlled laboratory measurements in a vacuum chamber. But the working standards of the kilogram will be disseminated—physically delivered to measurement-science laboratories—in the form of metal masses in the open air. That means that water vapor and whatever else is in the air can adsorb onto the surface of a kilogram working standard, causing inaccurate measurement of its mass.

Because humidity and air contaminants differ substantially around the world, measurements of a carefully calibrated mass standard can differ appreciably from place to place at the levels of accuracy needed for industrial and scientific metrology. If, however, a calibrated QCM were to accompany each standard, it could provide an accurate measure of the amount of material adsorbed in transit and at the destination, helping the labs to receive more accurate definitions of the new kilogram while taking environmental conditions into account.

More information: C Stambaugh et al, Linking mass measured by the quartz crystal microbalance to the SI, *Metrologia* (2019). DOI: 10.1088/1681-7575/ab54a5

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