# Compact Sensor for Environmental Monitoring

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This report describes a simple means of detecting trace amounts of gaseous substances in the atmosphere. In the fabrication processes for semiconductor LSIs, production defects occur if gas phase contaminants in the atmosphere stick to the wafer being processed. To overcome this problem, I have developed and am currently applying a contamination sensor that uses a quartz crystal microbalance (QCM) to detect such contamination. The detection system using this sensor has high sensitivity and is well suited to tracking the time-varying concentration of an atmospheric contaminant. Investigating the behavior of contaminants in a semiconductor LSI fabrication plant makes it possible to identify contamination sources and eliminate them. Therefore, this sensor system should be effective in lowering production costs and improving product quality. Moreover, the sensor is compact, so it can be easily installed, for example, at various locations around a plant and in the wafer container. Besides being compact, the QCM controller is easy to use and offers outstanding portability: it simply needs to be plugged into the universal serial bus (USB) port of a device such as a notebook computer. As a result of these features, it is likely that this sensor will not only be used in semiconductor fabrication plants but also be applied to other kinds of environmental measurement.

# 1. Introduction

As the degree of integration of semiconductor LSIs increases, it is becoming more important to control the environment surrounding a wafer. When gaseous contaminants from the atmosphere adhere to a wafer being processed, they can become the cause of fabrication defects. With denser integration, the sensitivity to such chemical contamination also increases. The International Technology Roadmap for Semiconductors (2007 Edition)<sup>1)</sup> clearly states that there is a growing need for affordable, accurate, repeatable, real-time sensors for nonparticulate contamination.

Nowadays, wafer containers, such as the front-opening unified pod (FOUP) and the standard mechanical interface (SMIF) pod, are commonly used to create "clean" local environments around wafers. However, they do not eliminate the problem of chemical contamination from the ambient atmosphere. In fact, since contaminants become trapped in a closed space like a wafer container, analysis of the wafer environment is difficult. Consequently, with the aim of developing a sensor for analyzing the wafer environment, I have focused my attention on the quartz crystal microbalance (QCM). When a substance is adsorbed onto the crystal surface of the QCM, the mass-and, in turn, oscillation frequency-of the resonator changes. By measuring this frequency change with a frequency counter, one can determine the mass of the adsorbed substance. In other words, the principle of the QCM is to use the quartz resonator as an adsorption-type atmospheric sensor.

Generally, to analyze an atmosphere, one first aspirates the atmosphere with a suction pump to concentrate the contaminant in question. After that, one applies ion chromatography or mass spectrometry coupled with gas chromatography. With this method, not only does the sampling take time, but the sample must be transferred to the location of the analysis apparatus. As a result, it is a long time before the analysis results are available. In comparison, the QCM sensor—whose key features are listed below—is convenient for contamination analysis.

- 1) Measurement in real time is possible.
- 2) High sensitivity can be obtained.
- 3) An inexpensive measurement system can be configured.
- 4) The absence of moving parts means that particle generation is not a concern.
- 5) The sensor is compact, so it can be inserted into small spaces like wafer containers.

In this paper, I explain the principle of the QCM, describe my QCM, and present some example measurement results.

## 2. Principle

In 1959, Sauerbrey<sup>2)</sup> showed that the change in oscillation frequency when a substance adsorbs to the surface of quartz oscillator is given by

$$\Delta f = -\frac{f^2 \Delta m}{N \rho_Q F} ,$$

where  $\Delta f$  is the change in oscillation frequency due to the adsorbent, f is the oscillation frequency before adsorption of the contaminant,  $\Delta m$  is the change in mass due to the adsorbent, N is a frequency constant (0.167 MHz mm for AT cut crystal),  $\rho_Q$  is the density of the crystal oscillator (2.65 g cm<sup>-3</sup>), and F is the surface area of the oscillator. According to this equation, the variation in oscillation frequency is proportional to the adsorption amount. If a quartz oscillator with oscillation frequency of 25 MHz is used,  $\Delta f$  of 1 Hz will be equivalent to an adsorption amount of 1 ng cm<sup>-2</sup>. Since a frequency counter with stability to a resolution of nine decimal places can easily be constructed, it is possible to detect sub-nanogram amounts of adsorbents with a 1 cm<sup>2</sup>-sized QCM.

## 3. Apparatus

A photograph of the developed QCM system is shown in **Figure 1**. The controller is compact  $(120 \times 100 \times 40 \text{ mm}^3)$  and light (total weight: 390 g), and it is used by simply connecting it to the universal serial bus (USB) port of a portable electronic device like a notebook computer, which also provides the power to run the QCM. The sensor unit (which uses the quartz crystal as a sensor) is connected to the controller via a 3-mlong cable. Up to five sensor units can be used simultaneously.

I prepared two types of sensor units (suitable for different setups). One type was set up indoors in the manner shown in Figure 1. The other type, shown in **Figure 2**, is only 2.5 cm in diameter and 9 mm thick, so it can be inserted into a small closed space such as a wafer container even when it is fully loaded with wafers. For internal and external connections, it has a flexible cable (thickness: 0.1 mm, width: 6 mm, length: 3 cm), which passes through the lid



Figure 1 Appearance of QCM system.



Figure 2 Sensor for closed environment.

of the main body of the wafer container. There is no need to modify the wafer container, and the cable is thin enough that it has little effect on the sealing performance of the container's O-ring.

# 4. Example measurements

#### 4.1 Measurements in a plant environment

Below, I give two examples of measurements in a plant in areas in which chemicals are and are not used.

1) Measurements in an area in which chemicals are often used

The results of measurements taken over 50 days by sensors set up in two areas in which chemicals are often used (rooms in buildings A and B) are shown in Figure 3. Sensors with two different surfaces, silver and gold, were used. The measurements taken in these two buildings are plotted as lines (1) and (2), respectively. The monotonic increases for both lines indicate that the amount of adsorbent increased with time in both rooms. In the case of the sensor with a silver surface, the adsorption rate, which was calculated from the slope of the adsorbent-amount lines, was the same in both buildings, namely,  $40 \text{ ng cm}^{-2} \text{ day}^{-1}$ . From this result, we can infer that the chemicals used in these buildings were adsorbed over the measurement period. Looking



(a) H<sub>2</sub>S concentration measured outside the plant. Based on data published by local government body.
(b) Signals from QCM set up in plant.

#### Figure 3 Example measurements taken in plant.

at both lines closely reveals sudden rises all along the lines. Since these sudden rises occur for both buildings at the same time, we suspect that something must have intruded into the rooms from outside the plant. The concentration of hydrogen sulfide (H<sub>2</sub>S) measured outside the plant (extracted from atmospheric-monitoring data collected by a local government agency) is plotted in Figure 3 (a). Comparing the  $H_2S$ data plot with the two adsorbent-amount lines (1) and (2), we see that the peaks in the lines coincide in time with the H<sub>2</sub>S concentration peaks. Since H<sub>2</sub>S has low solubility in water, it is hardly eliminated at all by the air washers on the outside-air-intakes of the plant buildings, and it manages to intrude into the plant. In this factory, even though countermeasures are taken to prevent  $H_2S$  coming into contact with manufactured goods, and the H<sub>2</sub>S intrusion thus does not cause any problems, attention should be paid to intrusions from the outdoor air.

On the other hand, the variation in adsorbent amount measured by a sensor with the gold surface is plotted as line (3) in Figure 3 (b). This line is less steep than those for the silversurface sensors and it has no peaks. Thus, it is possible to narrow down the detection targets to a certain extent by selecting the material of the sensor surface.

2) Measurements in an area in which chemicals are not used

Measurements taken over a few days in the vicinity of thermal-processing equipment in an area in which chemicals are not used (in the same factory as above) are plotted in **Figure 4** (a). Plot (1) shows measurements taken near the upper part of the equipment, and plot (2) shows ones taken near its lower part. As in the first example, the adsorbent amount increased monotonically with time. However, in this case, the rates of adsorption were 14 and 7 ng cm<sup>-2</sup> day<sup>-1</sup> for lines (1) and (2), respectively. Since these values are smaller than those in the previous



Figure 4

Results of measurements taken by sensor set up near thermal-processing equipment.

two examples, it can be said that this building environment is the cleaner of the two examples because no chemicals are used. A close look at the adsorbent-amount lines reveals numerous small variations, or spikes, along both lines. The initial time period of Figure 4 (a) is expanded in Figure 4 (b). The variation in the furnace temperature of the thermal processing equipment (obtained from operation records) is plotted against time in Figure 4 (c). A comparison of lines (1) and (2) in Figure 4 (b) with the line in Figure 4 (c) reveals that when the adsorbent amount recorded by the sensor near the equipment's upper side [line (1)] increased, the furnace temperature simultaneously increased. This increase in adsorbent amount is explained by the fact that hydrocarbons accumulated on the equipment and bits of its paint coating were detached by the heat from the furnace and emitted when the furnace was fired up.

Moreover, the increase in adsorbent amount recorded by the sensor near the equipment's lower side [line (2)] corresponds to the time when the hot product was taken from the furnace just after thermal processing. This result is also explained by materials being emitted as a result of the furnace's heat.

Although this sensor is unable to identify the adsorbent, a major advantage of this sensing method is that it lets one obtain information concerning the origin of the contaminant in real time by checking against other analytical results, operational records, and so on.

#### 4.2 Measurements in a closed space

To analyze the ambient atmosphere, one generally samples the air and collects substances in the air on an absorbing agent. It is difficult to apply that procedure to atmospheric analysis of a closed space like a wafer container. However, the QCM is compact enough that it can be inserted into a wafer container and used to analyze the container's atmosphere. Some example measurements taken by the closed-space sensor



Figure 5 Results of measurements taken by sensor placed inside wafer container.

shown in Figure 2 inserted into a wafer container are plotted in Figure 5. Two wafer containers were used (after being cleaned). One container (a) had a wafer placed in it immediately after chemical processing; the other container (b) was left empty. A QCM sensor was then inserted into each container and used to measure the container's internal atmosphere. In container (a), the absorbent amount increased with time; however, in container (b), it remained unchanged. This result can be explained as follows: the chemicals used during wafer processing remained on the surface of the wafer and were subsequently emitted into the wafer-container atmosphere. The decrease in the slope of the graph [case (a)] with time indicates that the emission amount gradually decreased.

Although this sensor does not enable qualitative analysis, if surface analysis by methods such as X-ray photoelectron spectroscopy and secondary ion mass spectroscopy is carried out on the sensor surface after it has been used, information complementary to the QCM data can be obtained. (In practice, a clean metal sheet is placed next to the sensor beforehand and analyzed instead of the actual sensor surface.) However, although this procedure consistently provides complementary information, the key merit of the QCM sensor is that it can provide us with knowledge about an ambient atmosphere in real time even in a small closed space.

## 5. Conclusion

A technology (QCM sensor system) that uses a standard device (quartz oscillator) to detect chemical substances in an ambient atmosphere with high sensitivity in real time has been developed. Although the QCM is a useful tool, it has the drawback that it cannot really perform qualitative analysis. With that drawback in mind, and also wishing to give the QCM high sensitivity and selectivity, I will try to develop the QCM as a sensor for use in ordinary environments This technology has been licensed to Takane Electronics Co., Inc., under the Intellectual Property Strategy Implementation Project administered by Kawasaki City Council, and is currently being readied for commercialization.<sup>3)</sup>

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