Real time monitoring system of Airborne Molecular Contamination in the clean room

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Abstract: We developed sensitive real time molecular contamination monitoring system for semiconductor clean room using Quartz Crystal Microbalance (QCM) technique. We can successfully observe the daily variation of molecular contamination density in the clean room and simultaneous emission phenomena with operation sequence of production equipments. Systems are composed of 5 unit sensor head with 10m-long cable and computer for control and data storage. Sensitivity of QCM was calibrated by deposition of Si thin films on quartz crystals.

INTRODUCTION

The minimum future size of High-end Integrated Circuits becomes lower than 100 nm. And introduced many new materials to improve performance. Cu wiring and organic or porous SiO_2 isolation layer are under development. Those materials are very sensitive for molecular contamination. But it is very difficult to find out the occurrence of contamination troubles. Because, those troubles are unable to detect by usual in-line defect detection systems but they are only able to find out by final electrical test which is carried out several weeks later. But at that time, in most of the case, there are no evidences which happened while process were performed. So, it is very difficult to investigate the cause and origin of contamination troubles. Then, if it is possible to able to monitor the environmental molecular contamination condition of wafer process during 24 hours, it is very effective to investigate and identify the cause of contamination troubles. So, We developed compact and simple but sensitive molecular contamination monitoring system for semiconductor manufacturing clean room using Quartz Crystal Microbalance technique.

1. SYSTEM DESIGN OF QCM MONITOR

To evaluate daily variation of molecular contamination condition in the clean room and find out the emission events from production equipments, we developed compact sensor head with 10m long signal transmission cable and five-channel QCM monitor system. Monitor system were designed in consideration for following three points,

(1) This monitor system should be able to measure multi-point data on same time to evaluate two-dimensional density distribution and flow of molecular contamination vapor.
(2) Size must be compact which can set everywhere without disturbance for the operation of equipments and also minimize the disturbance for maintenance work.
(3) System cost should be minimized.

QCM sensor crystal and oscillator circuit are set in the metal case shaped with 30mm diameter and 45mm long. At the top of sensor case, metal mesh is mounted to enter the measuring vapor and eliminate electrical noise. The oscillation signal is amplified and transmitted through 1.5D-2V coaxial cable to monitor system. The monitor system is designed to considering for thermal stability and toughness for electrical noise to ensure the signal reliability. The system is made up of large scale one chip Field Programmable Gate Array (FPGA) and analog input buffer amplifier. Time base X'tal oscillator is selected TCXO with thermal stability less than 10ppm. The system is controlled through RS-232C interface to keep the system stability which is set among high power consumption production equipments. The monitor system and sensor head are shown in Fig.1 and 2

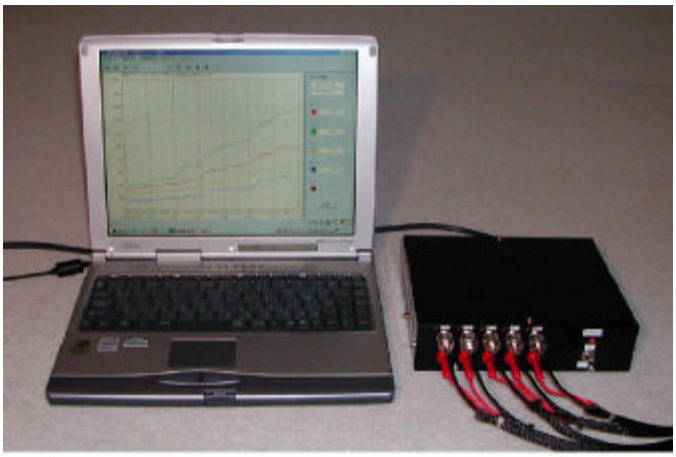

Fig.1 QCM Real time molecular contamination monitoring system and control & data logging computer. System size is 19.5cm(W)×16cm(D)×5cm(H).

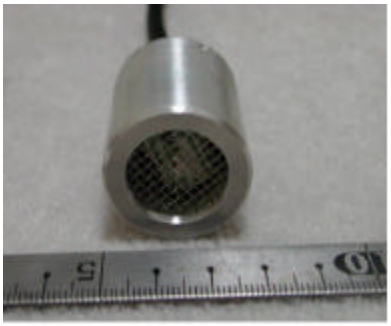

Fig.2 QCM sensor head. Size is 30mm (Diameter) and 45mm long (Typical Head)

Data sampling and filing system are controlled by calendar and clock of controlling computer. Measurements execute every 10 sec and averaged them every 3min. Data file is created at the time of 0:00 named "YYMMDD.XTM" (Y: Year, M: Month, D: Date) and closed at 23:57 after the end of the last measurement of the day. The number of data points are 480 points per day and data size is about 38~39 KB per day with comments word. Monitoring data is displayed graphically last 22 to 24 hours trends and also read latest numerical value.

2. QCM SENSOR CALIBLATION

The sensitivity of QCM sensor is reported as follows by [1], [2]

$$\delta m = -\delta f \cdot (v_s \cdot S_e \cdot \rho_q) / f_0^2 \quad \cdots (1)$$

Where, δm: adsorbed mass per unit area, δf: amount of frequency shifts, v_s : sound velocity in the quartz (5.72×10^5 cm/sec), ρ_q : density of quartz ($2.65 g.cm^{-3}$), and Se : area of electrode formed on quartz crystal.

According this theory , for 25MHz crystal with 3.5mmϕ electrode, the calcurated sensitivity become 0.23 ng /cm^2/Hz .

We calibrated sensisitivity of crystals by deposition of Si thin films on both side of them. Si film deposition was done by high vacuum electron beam evaporation system. Film thickness was measured by INFICON Model XTM/2 deposition monitor. Sensor crystals were washed in an organic solvent with ultra sonic bath and dried by nitrogen gas brow. After washing, initial oscillation frequencies were measured soon. Then samples were deposited about 20nm thick non-doped Si films on both sides. And oscillation frequencies were measured again as soon as film deposition. Amount of frequency shifts were shown in Table 1.

Table 1. Frequency shifts measurements before and after Si film deposition.

Specific gravity of Si =2.33g/cm3
Weight of 1nm Si film = 2.33×10-7 g/cm2 =233ng/cm2

Si Deposition

1st layer	2nd layer	Total thickness	Total weight
21.3nm	20.0nm	41.3nm	9622.9ng/cm2

	Initial freq.	Just after depo.	Δf	Sensitivity
Lot1-1	25021171.83	25011196.96	9974.87	0.96 ng/hz
Lot1-2	25024626.55	25014618.41	10011.14	0.96 ng/hz
Lot1-3	25019202.10	25009234.71	9977.39	0.96 ng/hz

Si Deposition

1st layer	2nd layer	Total thickness	Total weight
20.6nm	20.8nm	41.4nm	9646.2ng/cm2

	Initial freq.	Just after depo.	Δf	Sensitivity
Lot2-1	24979864.75	24969863.28	10101.47	0.95 ng/hz
Lot2-2	25005848.15	24995856.50	10091.65	0.96 ng/hz
Lot2-3	24986894.15	24985843.00	9891.16	0.97 ng/hz

From these experiments, we can show good reproducibilities and get the seisitivity of 0.96ng/cm^2/Hz for 25MHz sensor crystals. These values are as small as about 1/4 of theoretical value.

3. ADSORPTION CHARACTORISTICS OF SENSOR

We measured adsorption characteristics of Iso Propyl Alcohol (IPA) and water vapor on deferent surface sensor. Experimental atmospheres are prepared by controlling the mixture rate of carrier nitrogen gas and sample gases. The sample gases are prepared by nitrogen gas bubbling through IPA and purified water respectively. Two QCM samples are mounted parallel to each other separated with 6mm distance and set at the end of the small aluminum gas chambers. The cross section of gas chamber is 4cm×4cm and amount of total gas flow is controlled with 1 litter per minutes to keep the gas flow velocity to 1cm per sec.

Fig.3 and Fig 4 shows IPA adsorption characteristics on Au and Si surfaces.

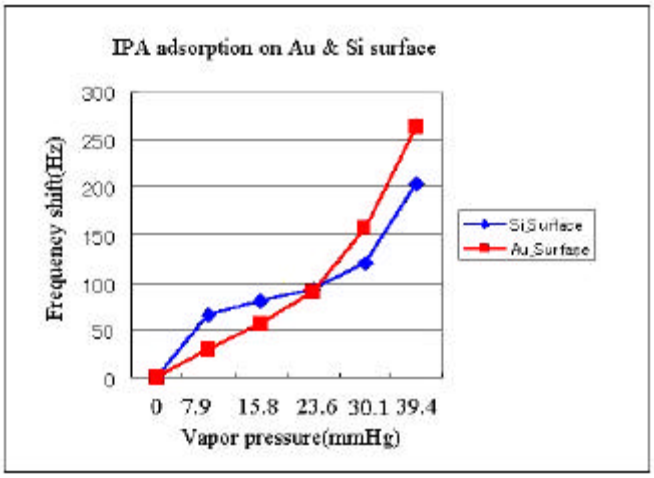

Fig.3 Adsorption characteristics of IPA vapor pressure and frequency shifts of Au and Si surface sensors.

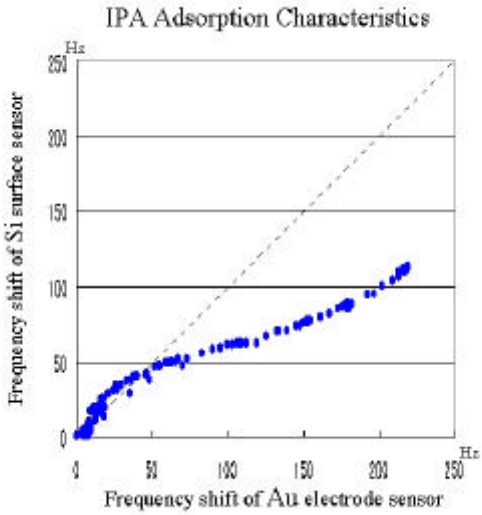

Fig.4 IPA adsorption characteristics on Au & Si surfaces.

These data show that the amount of adsorbed IPA molecules on Au and Si surfaces increase as vapor pressure increases more. Especially at low pressure region, IPA molecules adsorb on Si surface more than Au surface. On the other hand water molecules adsorbed on Au and Si surface in proportion to relative humidity at 23 ℃. (Fig.5) But adsorption coefficient of Si surface is about seven times greater than that of Au surface.

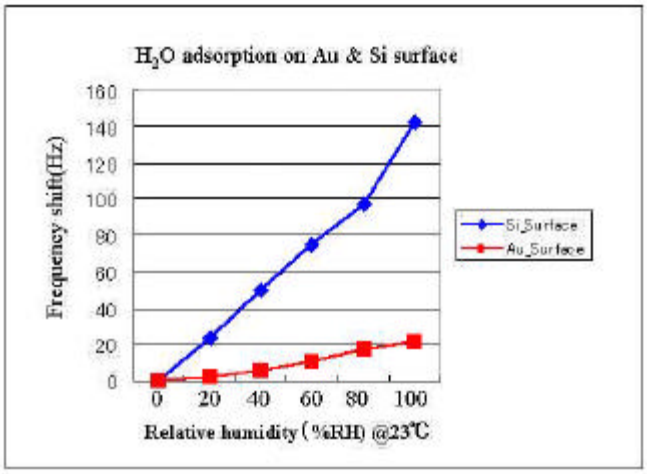

Fig. 5 Water molecule adsorption characteristics on Au and Si surface.

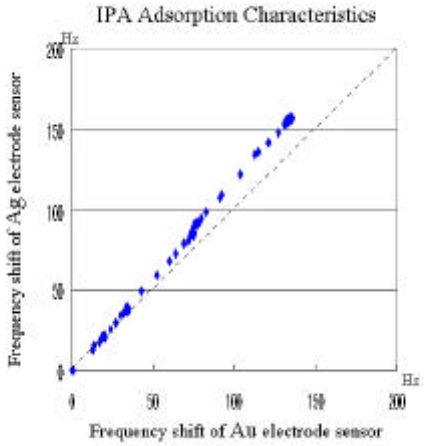

Fig. 6 IPA adsorption characteristics on Au and Ag surface

Fig.6 shows IPA adsorption characteristics on Au and Ag surface. In the dry nitrogen atomospher condition, IPA molecules adsorbed on Au are almost same amounts as that done on Ag surface.

4. FIELD EXPERIMENT

Fg.7 shows the one of the experiment data for the long time compared electrode quartz crystals of Au with that of Ag. This data shows the trend of observation for one week from June 23 to June 29 of 2002. According to our experiments in dry nitrogen atmosphere, the sticking coefficient of IPA molecule on Au and Ag surface seems to be same. But monitoring for long time in the usual air condition shows that Ag electrode sensor has more than six times higher sensitivity than Au electrode sensor. This effect may be due to the difference of chemical activity of Au and Ag, and existence of water molecules.

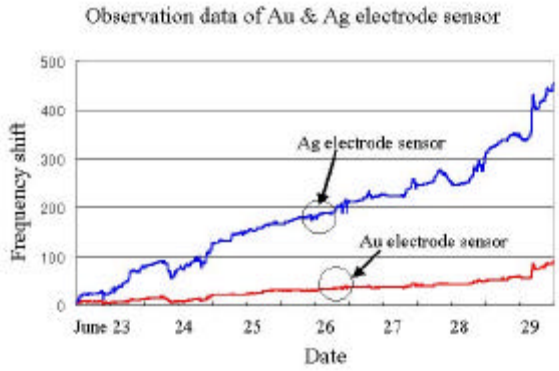

Fig.7 Frequency shifts data of Au and Ag electrode sensor in usual air condition. Those sensors were set at same position.

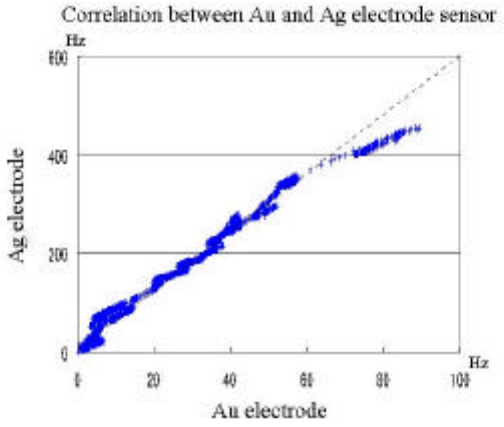

Fig.8 The correlation of Au and Ag electrode sensor in monitoring at usual air condition.

Fig.8 shows correlation analysis of Fig.7 data. The overall Au and Ag electrode have good correlation, but there are many fine variations in them.

Fig.9 shows frequency shift trend and its correlation from 0:00 to24:00 at June 24 which shown in Fig.7. The data concentrate to two sharp lines. The data group from 0:00 to 11:00 is concentrated in lower line and data form 12:00 to 24:00 is concentrated in upper line. At only one hour between from 11:00 to 12:00, correlation curve shifts suddenly more than 10 Hz. This phenomenon may be suggest to the presence of corrosive gas like H_2S. The Ag electrode surface slightly tarnish to dark brown after two week observation. Using the correlation analysis between Au or

Si to Ag or Cu surface sensors, it is able to distinguish organic contamination from corrosive contamination.

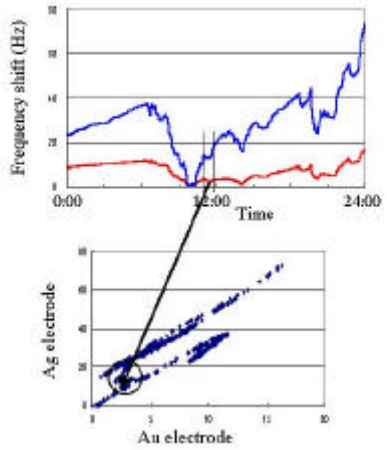

Fig.9 The frequency shifts trend graph and correlation between Au and Ag electrode sensor during time of 0:00 to 24:00 of June 24 data shown in Fig.7. The correlation curves of Ag electrode suddenly shift more than 10 Hz between 11:00 to 12:00.

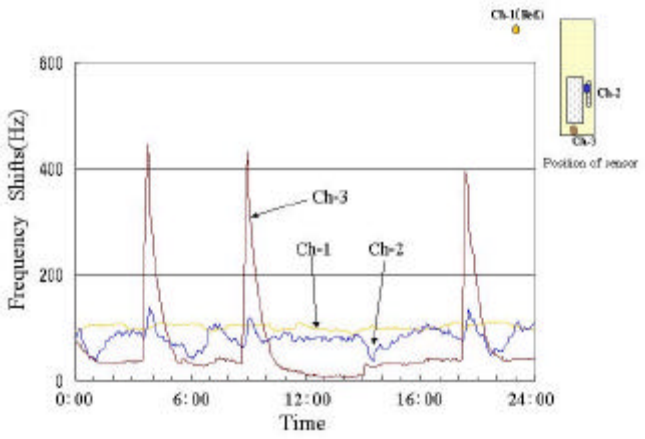

Fig. 10 Examples of QCM signals set around thermal CVD furnace.

Fig. 10 shows the signals set around CVD furnace. Each signal shows good correspondences with process sequence. But if examined them in details, the height and fine shape slightly differ in each sequences. These differences show which there are some fluctuations in process or environmental condition.

5 SUMMARY

We developed compact multi-channel QCM system for monitoring molecular contamination in semiconductor clean room. We verified the sensitivity of 25MHz quartz sensors by deposition of Si thin films and achieved less than 1ng/cm^2/Hz. We can successfully demonstrate the abilities and effectiveness of QCM system to observe the fluctuation phenomena of molecular contamination condition in clean room atmosphere.

REFERENCES

[1] Sauerbrey. Zeitschrift fur physik, 155,206, 1959

[2] Chih-Shun Lu, Qwen Lewis. J.Appl.Phys, Vol.43 No.11. 1972