



## Real time evaluation of silicon epitaxial growth process by exhaust gas measurement using quartz crystal microbalance



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### ARTICLE INFO

#### Keywords:

Quartz crystal microbalance  
Silicon epitaxial growth  
Minimal Fab  
*in situ* monitor

### ABSTRACT

A silicon epitaxial growth process in a trichlorosilane-hydrogen system was evaluated using a quartz crystal microbalance (QCM) placed at the exhaust of a chemical vapor deposition reactor designed for the Minimal Fab. The QCM showed two types of the frequency decrease behaviors, that is, *i*) a quick shift due to the gas property change caused by the trichlorosilane gas introduction into the ambient hydrogen and *ii*) the continuous and gradual decrease due to the byproduct deposition on the QCM surface during the silicon epitaxial growth. Because both *i*) and *ii*) showed a relationship with the silicon epitaxial growth rate, the *in-situ* information obtained by the QCM was expected for the real time monitoring of the film deposition process.

### 1. Introduction

Silicon epitaxial growth has a long history [1,2] in the electronics industry. Its industrial processes use chlorosilane gases, such as trichlorosilane ( $\text{SiHCl}_3$ ) and dichlorosilane ( $\text{SiH}_2\text{Cl}_2$ ) [3–5]. The produced films are uniform and flat at the atomic level and the growth rate is quite high.

Due to its reasonable cost and ease of use, the trichlorosilane gas has been very often used. The trichlorosilane gas, having the boiling point of 31 °C, is produced by the bubbling technique, *i. e.*, injecting hydrogen gas into the liquid trichlorosilane contained in a cylinder. In a mass-production plant having many epitaxial reactors for large diameter wafers, a huge amount of the trichlorosilane and hydrogen gas mixture is produced at the precisely controlled concentration and is supplied from a large tank to the reactors *via* tube networks. In contrast, in very small equipment, such as the Minimal Fab [6,7] using small wafers of a half-inch diameter, the gas supply condition is quite different from the mass-production plant. The compact-designed Minimal chemical vapor deposition (CVD) reactor [8–10] consumes significantly small amounts of the precursors in a short time period, for example, the trichlorosilane gas at the flow rate of 10–20 sccm at atmospheric pressure for a few minutes. In order to finely control the trichlorosilane gas concentration at the significantly low gas flow rate, any *in situ* real time measurement method is helpful. Additionally, the measured information is expected

to show the relationship with the epitaxial growth rate.

For this purpose, a piezoelectric crystal microbalance, such as a quartz crystal microbalance (QCM) and a langasite crystal microbalance [11–18], may be a candidate, because it can sensitively detect the significantly weak signals such as the change in the gas properties and the increase in the nano-gram-level weight of the thin film formed at the surface. Previous studies [14–17] have reported that the deposition of the byproducts of  $(\text{SiCl}_2)_n$  from the trichlorosilane gas could be *in situ* measured by the QCM. The byproduct formation and deposition measured by such sensors should be evaluated as to whether they have a relationship with the epitaxial growth rate. Such the relationship will help developing and controlling the epitaxial growth process.

In this study, the QCM was used to measure the exhaust gas of the silicon epitaxial reactor designed for the Minimal Fab. The typical QCM frequency changes were, for the first time, classified and evaluated for obtaining the *in-situ* real time information of the epitaxial growth process.

### 2. Experimental procedure

Fig. 1 shows the Minimal CVD reactor and the QCM system. This reactor consists of a half-inch silicon wafer (12.5-mm diameter and 0.25-mm thick), a transparent quartz tube, a wafer holder made of

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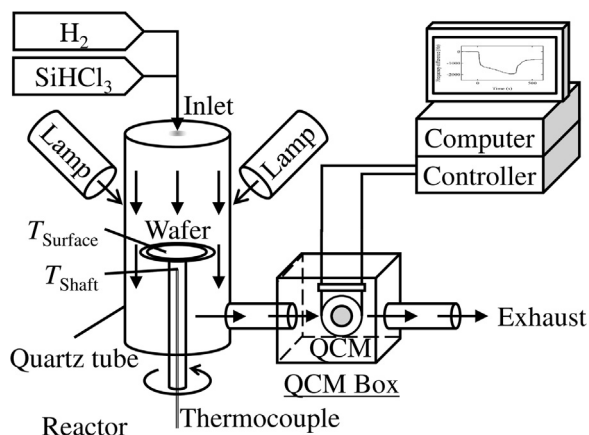


Fig. 1. Chemical vapor deposition reactor for Minimal Fab., having a quartz crystal microbalance.

quartz glass, a gas inlet and three heating units consisting of a halogen lamp and a reflector. The inner diameter of the quartz tube is 24 mm. The wafer holder diameter is 19 mm. The wafer is rotated at the rate of 0–30 rpm. The infrared light emitted from the halogen lamps for the heating is concentrated on the half-inch wafer surface. The quartz tube wall is cooled from the outside by flowing air [6] in order to maintain the cold wall environment.

The precursor gas and the carrier gas, trichlorosilane and hydrogen, respectively, are introduced at atmospheric pressure from the top of the reactor through the gas inlet. The hydrogen gas flow rate was 85 sccm, a part of which was divided and injected into the liquid trichlorosilane cylinder for the bubbling and for producing the gas mixture of trichlorosilane and hydrogen. The trichlorosilane gas of typically 21 sccm at 25 °C was produced by the hydrogen gas at the flow rate of 35 sccm, based on the trichlorosilane vapor pressure [19]. The total gas flow rate was typically 106 sccm. The trichlorosilane gas concentration significantly depends on the liquid temperature.

The trichlorosilane gas causes the surface reactions based on Eqs. (1) and (2). The symbol \* indicates the species chemisorbed at the surface. The rate equation [20] is written as Eq. (3) following the Eley-Rideal model.



$$R_{\text{Si}} = \frac{k_{\text{ad}} k_r [\text{SiHCl}_3] [\text{H}_2]}{k_{\text{ad}} [\text{SiHCl}_3] + k_r [\text{H}_2]} \quad (3)$$

$R_{\text{Si}}$  is the silicon epitaxial growth rate.  $[i]$  is the concentration of species  $i$  at the wafer surface.  $k_{\text{ad}}$  and  $k_r$  are the rate constants of Eqs. (1), (2), respectively.

A significantly small amount of  $* \text{SiCl}_2$  desorbs from the silicon surface into the gas phase for producing  $\text{SiCl}_2$ , following Eq. (4) [17]. Even in the cold wall environment, the trichlorosilane gas produces a significantly small amount of  $\text{SiCl}_2$  by thermal decomposition in the gas phase, as described by Eq. (5). The  $\text{SiCl}_2$  finally produces  $(\text{SiCl}_2)_n$ , as denoted by Eq. (6), in the gas phase and at the solid surface. The QCM sensor is expected to detect the deposition of  $(\text{SiCl}_2)_n$  on its surface by means of measuring the significantly small weight increase at the ng/cm<sup>2</sup> level.



The voltage applied to the halogen lamps was fixed at 80 V. Because the trichlorosilane gas absorbs a part of the infrared light emitted from

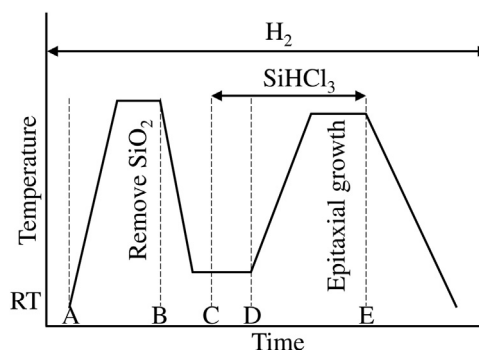


Fig. 2. Silicon epitaxial growth process used in this study. A: heater on, B: temperature decrease, C: initiate trichlorosilane supply, D: temperature increase and E: terminate trichlorosilane supply and heater off.

the halogen lamps [9,21], the gas phase temperature, and consequently the wafer temperature, increased with the increasing trichlorosilane gas concentration. For monitoring the thermal condition, the temperature at the top of the wafer rotation shaft,  $T_{\text{Shaft}}$  (°C), shown in Fig. 1, was measured using the R-type thermocouple. The  $T_{\text{Shaft}}$  value was considerably different from the wafer surface temperature,  $T_{\text{Surface}}$ , shown in Fig. 1 due to the distance between them [9].

The QCM sensor (25 MHz, Hallolan Electronics Co., Ltd., Tokyo) was installed at the exhaust, as shown in Fig. 1, for avoiding the metallic contaminations due to the QCM sensor consisting of various metals. The exhaust gas entered into the QCM box, then passed over the QCM sensor surface. The QCM frequency was obtained by the controller and was recorded by a personal computer. The QCM is expected to obtain the significantly weak information [14–16] related to the silicon epitaxial growth process such as the gas property change and the by-product deposition. Although the measurement at the exhaust might contain the results by various processes in the reactor, the major trend was expected to be obtained even from the scattering data.

Fig. 2 shows the process used in this study for clearly observing the QCM frequency behavior by separating it from the temperature influence. During Steps A–B, the wafer was heated to a high temperature for cleaning the silicon wafer surface. During Steps B–C, the temperature was decreased so that the trichlorosilane did not cause any chemical reaction. During Steps C–E, the trichlorosilane gas was introduced into the reactor. By this sequence, the trichlorosilane introduction could be clearly detected by the QCM frequency shift. During Steps D–E, the wafer temperature was increased by means of increasing the halogen lamp voltage in order to initiate the silicon epitaxial growth. Two minutes after increasing the halogen lamp voltage, the epitaxial growth was automatically initiated by the high temperatures. After maintaining Step D for an additional one minute in order to produce the silicon epitaxial film, the wafer was cooled at Step E. At the same time, the trichlorosilane gas supply was terminated. After taking out the wafer from the reactor, the epitaxial film thickness was evaluated by measuring the increase in the wafer weight and the wafer thickness.

Because the exhaust pipe (1/4-in. diameter) between the quartz tube and the QCM box was sufficiently long and thin, about a 0.5-m length and 0.25-in. diameter, respectively, the exhaust gas was sufficiently cooled to room temperature. Thus, any temperature change around the wafer did not influence the QCM frequency.

As reported in a previous study [13], the QCM frequency change is proportional to the product of the gas density,  $\rho$ , and gas viscosity,  $\mu$ , to the 1.3-th power,  $(\rho \mu)^{1.3}$ . As shown in Fig. 3, the  $(\rho \mu)^{1.3}$  value is nearly proportional to the mole fraction of the trichlorosilane gas in ambient hydrogen at room temperature and at atmospheric pressure. Based on Fig. 3, the QCM frequency change corresponds to the increase and decrease in the trichlorosilane gas concentration.

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