Particle Counting in Semiconductor Processing Gas and Apparatus with a New Flow-Cell Type Laser Particle Counter

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A new flow-cell type laser particle counter has been developed in order to measure particles at low and high pressure or in flammable and toxic gases. The minimum detectable particle diameter was 0.17μ m. The helium leak rate of this particle counter was lower than 2×10^{-11} atm·cc/sec by using double O-ring seal structure. We have successfully measured particles in CVD apparatus at low pressure and in SiH4 gas.

1. Introduction

The yield of products in the manufacture of VLSI is strongly affected by particle contamination in production apparatus and in processing gases. In the conventional optical particle counters, sample air is drawn from an aerodynamical nozzle and a light beam crosses the sample air stream.⁽¹⁾⁽²⁾ Particles in the sample air are usually detected by a light scattering method. At high and low pressure, the air stream becomes a turbulent flow in the sensing volume and the particles stray in the sensor. Therefore, the particles cannot be measured accurately because of counting losses and erroneous counts. Furthermore. since the conventional particle counters have an incomplete seal structure and dead space, it is very difficult and dangerous to measure particles in the flammable and toxic gases.

In this paper, we report a new flow-cell type laser particle counter and the particle measurement in a CVD apparatus at low pressure and in semiconductor processing gases.

2. A New Flow-Cell Type

Laser Particle Counter

Figure 1 shows a schematic of the newly developed flow-cell type laser particle counter. The measured gas was introduced into a quartz flow-cell. A laser beam(output power: 40mW, wave length: 780nm and energy density in the flow-cell: 250W/cm²) was incident into flow-cell the and particles were detected by the light scattering method.(1) The minimum detectable particle diameter was estimated using a standard polystyrene latex(PSL) sphere in N₂ gas. The newly developed particle counter was able to detect 0.17µm PSL particles.

The double O-ring seal structure was used at the seal between the quartz flow-cell and the metal flange as shown in Fig. 2. The helium leak rate was lower than 2×10^{-11} atm·cc/sec by evacuating the sealing space between the two



Sealing space O-ring Exhaust port Flow-cell(quartz) VCR joint Metal flange(SUS316L)

Fig.2. Schematic of double O-ring seal.

Fig.1. Schematic of a new flow-cell type particle counter.

O-rings. When the pressure of the measured gas was below 1 atm, the sealing space was usually evacuated. On the other hand, when the pressure of the measured gas was higher than 1 atm, the pressure of the sealing space was kept a little higher than the pressure inside the flow-cell. Both the measured gas and the environmental air were protected to leak into the sealing space.

The flow-cell block has a commercially available joint such as VCR and MCG joints. The flow-cell block was able to be easily connected with a CVD or an RIE apparatus and with a gas supplying line. Furthermore, the optical system was able to be easily removed from a flow-cell block. The flow-cell block was able to be baked without removing the flow-cell block from the apparatus or the gas supplying line.

3. Results and Discussions

3-1. Particles in CVD apparatus at low pressure

Particles in a CVD apparatus was



Fig.3. Particle measurement in CVD apparatus.

measured at low pressure. The particle counter was connected with the CVD apparatus as shown in Fig. 3. N_2 gas was introduced to the reactor at 600sccm. The N_2 gas of 10sccm was sampled to the particle counter. The pressure in the reactor was controlled by the variable conductance valve. Figure 4 shows the



Fig.4. Detected particles after an open/close operation of the CVD apparatus. Pressure of the reactor, A:100Torr, B:45Torr.



Fig.5. Particle measurement in SiH₄ gases.

Table 1. Specifications of the advanced line and the conventional line.

	Cylinder	Filter	Pressure reducing regulator	
Advancd	Stainless	0.01µm	with diaphragm type	
line	steel	metal filter	pressure gauge	
Conventional	Manganese	7µm	with Bourdons type	
line	steel	metal filter	pressure gauge	

time variation of the number of particles after an open/close operation of the gate valve. The pressure of the reactor was (A) 100Torr and (B) 45Torr. After the gate valve operation, the number of particles increased and then decreased with time. Hence, particles induced by the operation of the gate valve was able to be detected at a low pressure with this particle counter.

3-2. Particles in SiH4 Gas

Figure 5 shows the experimental arrangement of measuring particles in SiH4 gases. Two types of SiH4 gases, 5% SiH4 in N2 and 10% SiH4 in H2, were supplied through a conventional and an advanced lines to the counter. The specifications of each gas lines are listed in Table 1. The dew-point of a purging Ar gas of both lines was below -95°C. No particles was detected in the purging Ar gas through both the conventional and the advanced lines. When the SiH4 gas was supplied from the conventional line to the particle counter. particles of 10²-10⁴ numbers/liter were detected. On the other hand, no particles was detected, when the SiH4 gas was supplied from the advanced line.

Filters are usually inserted in gas supplying lines to eliminate particles. However, reactive gases would generate particles by the reaction with residual moisture in the down stream of the filter. We measured particles in SiH4 gas, when the SiH4 gas was supplied from the different dew-point gas supplying line. Figure 6 shows the experimental arrangement. The SiH4 gas was supplied from the advanced line. The dew-point of the supplying gas was varied by switching the gas route(A) to route(B). The dew-point of purging Ar gas through route(A) was below -95°C and that through route(B) was -85°C. No particles in the Ar purging gas was detected both through route(A) and route(B). When the SiH4 gas was supplied through route(A) to the particle counter, no particles was detected. On the other hand, through route(B), many particles Table 2 summarizes the were detected. number of particles detected through route(A) and route(B). Consequently, we found that particles were generated at the down stream of the filter depending on the quality of the gas supplying line.

If the number of $0.17\mu m \phi$ particle is one million/cf (cubic foot: 1cf=28.3 liter), the impurity level corresponds to about 0.1ppb in mass ratio and about 0.1ppt in volume ratio. It is very difficult to measure such a low concentration impurity in gases using a conventional mass analyzer or gas chromatography. The particle counter using the light scattering method is significantly useful for detecting low concentration particles generated within a processing gas, e.g., SiO particles which are generated within SiH4 gas.

6. Conclusion

We have developed the new flow-cell type laser particle counter in order to measure particles at low and high pressure or in flammable and toxic gases. The new particle counter had leak tight structure using double O-ring seals. The minimum detectable particle diameter was 0.17µm. Particles in the CVD apparatus at low pressure and in the SiH₄ gas line have been successfully The new flow-cell detected. type



Fig.6. Measurement of particles in SiH₄ gas when dew-point was changed.

Dew-point**	Particle diameter and particle numbers (particles/liter)			
	>0.17µm	>0.20µm	>0.30µm	
Route(A) -95°C (37ppb)	0	0	0	
Route(B) -85°C (230ppb)	2000	220	0	

Table 2. Particle numbers in SiH4 gas.*

* SiH4 gas was supplied from the advanced line ** Dew-point of the purging Ar gas measured just before starting to flow the SiH4 gas.

particle counter is significantly useful for particle monitoring in VLSI production apparatus and processing gases.

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