

Effect of Gaseous Atmosphere on Particle Generation

by Emitters for Corona Discharge Ionizers

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A corona discharge air ionizer is one of the most useful devices for controlling electrostatic charges in ULSI manufacturing processes. However, a corona discharge air ionizer generates particles that act as contamination. Particle generation tests were carried out for a silicon emitter and a tungsten emitter in air, nitrogen and argon atmospheres. Deterioration at the tip of each emitter operated in each type of atmosphere was observed to investigate the relationship between the particle generation characteristics and the deterioration characteristics. The number of particles generated from the silicon emitter was smaller than that generated from the tungsten emitter in each type of atmosphere. The number of particles generated in argon and nitrogen atmospheres was lower than that in air. The silicon emitter in nitrogen and argon atmospheres generated about 200 times fewer particles than did the tungsten emitter in air. No deterioration was observed for the silicon emitter operated in nitrogen and argon atmospheres, while considerable deterioration was observed for the tungsten emitter operated in air.

1. Introduction

Phenomena related to electrostatic charge events, such as electrostatic discharge (ESD) and electrostatic attraction (ESA), cause serious yield reduction in the ULSI manufacturing process. An ESD event causes the breakdown of semiconductor devices, and an ESA event attracts particulate contamination onto the silicon wafers and reticles used in the lithography process. The corona discharge air ionizer has been widely used to prevent yield reduction in the ULSI production processes.

However, a corona discharge air ionizer generates particles that act as contamination on the semiconductor products ¹⁾. The particle generation characteristics depend on the properties of the emitter material when ionizer is operated in air ^{2 - 6)}, because an oxide film formed on the emitters has influence on the characteristics ^{7,8)}.

The jet ionizer has been proposed to operate the emitter in suitable gaseous atmosphere without oxygen $^{9,\ 10)}$. The ion

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generation in the jet ionizer conducted in a suitable gaseous atmosphere because the emitter and a grounded ring are installed inside the nozzle. Therefore, both particle generation and ion generation characteristics can be controlled by the kind of gas flowing inside the nozzle.

In this study, we investigated the particle generation characteristics and the deterioration characteristics of a silicon emitter and a tungsten emitter operated in air, nitrogen gas, and argon gas to find out the most suitable combination of emitter material and gaseous atmosphere for the jet ionizer.

2. Experimental setup

Figure 1 shows the setup for measuring the I-V characteristics, the particle generation characteristics, the ozone generation characteristics and the deterioration characteristics. The measurement of these characteristics was conducted for the silicon emitter and the tungsten emitter in air, and in nitrogen and argon atmospheres. The tungsten emitter was made of a high-purity (99.99%) tungsten rod, and the silicon emitter was made of a high-purity (eleven-nine) polycrystalline silicon rod. The spherical radius of the emitter tips was 0.3 mm. The emitter was located in a chamber (500 mm × 500 mm × 500 mm) made of acrylic resin and was connected to a steady 0-20 kV dc power supply. A metal ring with a radius of 50 mm made of copper wire of 2.0 mm diameter was placed 50 mm below the emitter tip as the grounded electrode and was connected to an ammeter to measure the discharge current. Air (40 % R.H.), nitrogen (less than 0.64 ppm H₂O) or argon (less

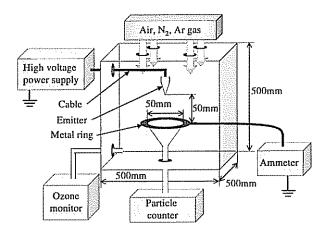


Fig. 1 Setup for measuring the I-V characteristics, ozone generation characteristics, particle generation characteristics and deterioration characteristics of an emitter in each type of atmosphere.

than 0.64 ppm H_2O) was injected into the chamber at the flow rate of 3.0 L/minute to replace the gas in the chamber.

The I-V characteristic, that is, the relationship between the emitter voltage V_E and the corona discharge current I_D , was measured to investigate the particle generation and emitter deterioration characteristics at the same corona discharge current. The particles generated from the emitter were measured with a particle counter with a particle size detection limit of $0.03~\mu$ m diameter. A glass funnel was placed 50 mm below the tip of the emitter to collect the particles. The number of particles counted was recorded continuously for 24 hours. The ozone generation characteristics were investigated by measuring the ozone density in the chamber using an ozone monitor. After 24 hours, the emitter tip was observed by a SEM (Scanning Electron Microscope) and analyzed by an AES (Auger Electron Spectroscope).

3. Experimental results

3.1 I-V characteristics

Figure 2 shows the relationship between the emitter voltage V_E and the corona discharge current I_D for the silicon emitter. In this figure, the threshold voltages depend on the type of the atmosphere. The threshold voltages in air, nitrogen and argon atmospheres are 8.5 kV, 9.0 kV and 6.4 kV for the positive polarity emitter and -9.0 kV, -8.6 kV and -3.8 kV for the negative polarity emitter, respectively. Figure 3 shows the I_D - V_E characteristic for the tungsten emitter. In this figure, the threshold voltages in air, nitrogen and argon atmospheres are 9.2 kV, 9.5 kV and 6.5 kV for

Table 1 Emitter voltage required for a corona discharge current of 0.5 μ A for each type of emitter in each type of atmosphere.

	Silicon emitter		Tungsten emitter	
	Positive	Negative	Positive	Negative
аіг	+9.2 kV	-9.3 kV	+9.6 kV	-9.6 kV
N ₂	+9.7 kV	-8.8 kV	+10.6 kV	-9.0 kV
Ar	+7.3 kV	-4.7 kV	+7.2 kV	-4.6 kV

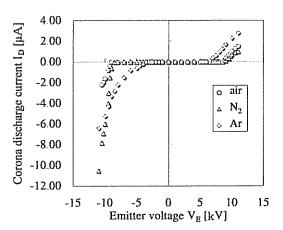


Fig. 2 I-V characteristics for the silicon emitter in each type of atmosphere.

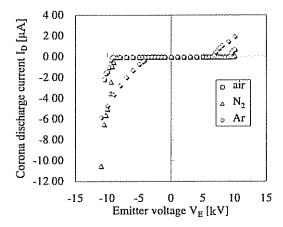


Fig. 3 I-V characteristics for the tungsten emitter in each type of atmosphere.

the positive polarity emitter and -9.2 kV, -8.5 kV and -3.9 kV for the negative polarity emitter, respectively. The I_D - V_E characteristic depends only on the type of atmosphere and is independent of the kind of emitter material.

The emitter voltage V_E used in the particle generation tests, ozone generation tests and deterioration tests was adjusted such that the corona discharge current I_D was $0.5\,\mu$ A because the corona discharge current I_D of $0.5\,\mu$ A is reasonable to prevent a spark discharge between the emitter and grounded ring in the jet ionizer. The values of emitter voltage used in these tests are summarized in Table 1.

3.2 Particle generation

The particle generation characteristics were measured for each emitter operated in each type of atmosphere. The cumulative number of particles N_P was counted for 24 hours without biasing the emitter in order to measure the background gas pollution. The cumulative number of background particles was 2 over a period of 24 hours. After that, each emitter was operated for 1 hour without counting to allow it to attain the steady state. Then the cumulative number of particles was measured for 24 hours for each emitter operated at the discharge current I_D of $0.5\,\mu\,\mathrm{A}$ in each type of atmosphere.

Figure 4 shows the cumulative number of particles N_P generated from the silicon emitter as a function of the operating time t_O for both the positive polarity emitter and the negative polarity emitter. Figure 5 shows the results for the tungsten emitter. In these figures, the cumulative number of particles generated from the positive polarity emitter is higher than that generated from the negative polarity emitter for both the silicon and the tungsten emitter.

The number of particles accumulated over a period of 24 hours is summarized in Table 2. In this table, the number of particles generated from the silicon emitter is much less than that generated from the tungsten emitter in each type of atmosphere. The number of particles generated in air is much higher than that generated in nitrogen and argon atmospheres independent of the kind of emitter material. In both figures 4 and 5, the number of particles generated by the positive polarity emitter is higher than that generated by the negative polarity emitter, when the emitter is operated in air.

3.3 Deterioration

Figures 6 and 7 show the SEM micrographs of the silicon and tungsten emitter tips. In these figures, (a) and (b) show the initial mirror-polished surfaces of the emitter tips. Micrographs (c) - (h)

Table 2 Number of particles generated from the silicon emitter and the tungsten emitter operated for 24 hours in each type of atmosphere.

	Positive	Negative	Average
air	87	68	77
N_2	8	7	8
Ar	3	3	3
air	475	394	435
N_2	106	85	96
Ar	82	64	73
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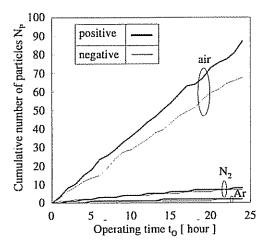


Fig. 4 Cumulative number of particles generated from the silicon emitter in each type of atmosphere.

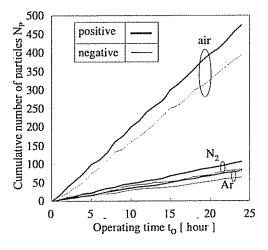


Fig 5 Cumulative number of articles generated from the tungsten emitter in each type of atmosphere.

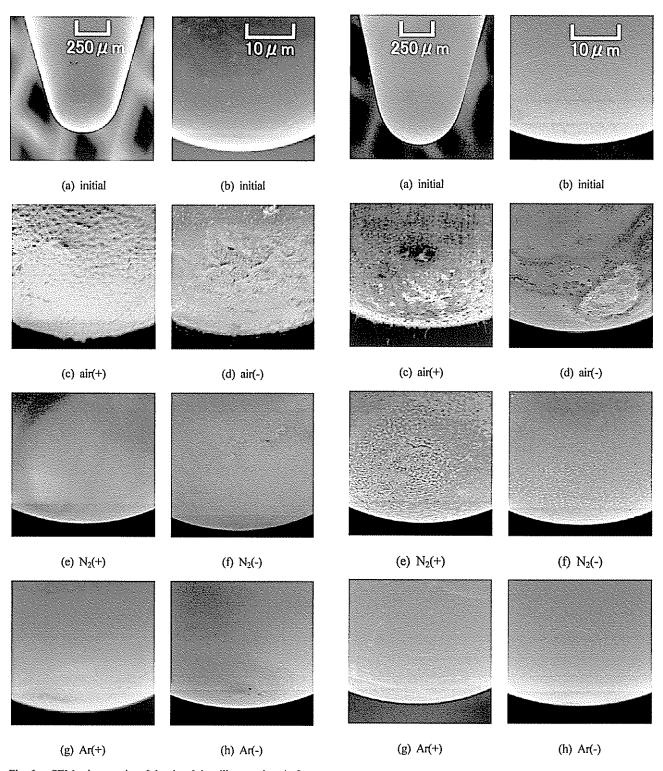


Fig. 6 SEM micrographs of the tip of the silicon emitter before and after operation for 100 hours in each type of atmosphere.

Fig. 7 SEM micrographs of the tip of the tungsten emitter before and after operation for 100 hours in each type of atmosphere.

show the emitter tips operated for 100 hours in (c), (d) air, (e), (f) nitrogen atmosphere, and (g), (h) argon atmosphere.

Figures 6 (c) and (d) show that the silicon emitter tips operated in air exhibit sandy surfaces because of deterioration. On the other hand, almost no deterioration is observed for the emitters operated in nitrogen atmosphere and argon atmosphere.

Figures 7 (c) and (d) show that the tungsten emitter tips operated in air exhibit considerable deterioration. On the other hand, the emitters operated in nitrogen atmosphere show slight deterioration. No deterioration is partially observed at the tips of the emitter operated in argon atmosphere.

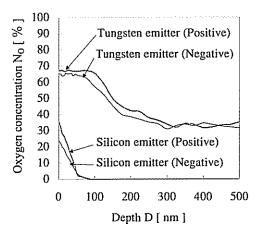


Fig. 8 Oxygen concentration at the tips of emitters as a function of depth for silicon and tungsten emitters operated for 100 hours in air.

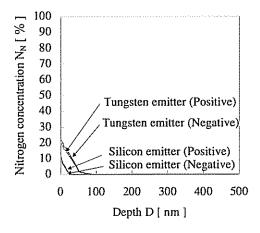


Fig. 9 Nitrogen concentration at the tips of emitters as a function of depth for silicon and tungsten emitters operated for 100 hours in air.

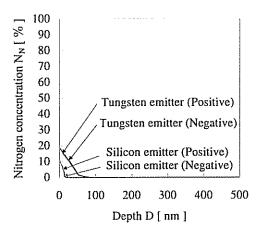


Fig. 10 Nitrogen concentration at the tips of emitters as a function of depth for silicon and tungsten emitters operated for 100 hours in nitrogen atmosphere.

3.4 Ozone generation and surface analysis of emitter tip

The ozone density in the chamber was measured during the operation in air. Ozone densities in the chamber were 0.014 ppm and 0.343 ppm for the operation of the positive polarity emitter and the negative polarity emitter, respectively.

The oxygen concentrations at the tips of silicon and tungsten emitters operated for 100 hours in air were analyzed by the AES. Figure 8 shows the oxygen concentrations for both the positive polarity emitter and the negative polarity emitter as a function of depth. A high concentration of oxygen was observed at the tungsten emitter tip. Figure 9 shows the nitrogen concentration at the tips of emitters operated in air. Nitrogen concentrations are 10 % and 20 % at the silicon emitter and tungsten emitter tips, respectively. Figure 10 shows the nitrogen concentrations at the tips of emitters operated for 100 hours in nitrogen atmosphere. Neither oxygen nor nitrogen was detected for emitters operated in argon atmosphere.

4. Discussion

The mechanisms of particle generation and tip deterioration are classified into two categories. One is sputtering of the emitter and the other is breakdown of the insulating thin film formed at the emitter surface. Sputtering would occur independently of the emitter material and atmosphere, while breakdown plays an important role only in the case of an emitter material that easily forms an insulating thin film.

In our experiments, a high concentration of oxygen was

observed at the tip of emitters operated in air, as shown in figure 8. This suggests that an oxide film is formed on the emitters. On the other hand, nitrogen concentrations of 20 % and 10 % were observed in the case of the tungsten emitter and the silicon emitter, respectively. These concentrations are insufficient to form an insulating nitride film. Therefore, only the emitters operated in air generate a great number of particles and show marked deterioration, because insulating films that cause breakdown are formed on these emitters.

The number of particles generated from the positive polarity emitter is higher than that generated from the negative polarity emitter, as shown in figures 4 and 5, and the deterioration of the positive polarity emitter tip is more marked than that of the negative polarity emitter tip, as shown in figures 6 and 7. These results do not agree with the results of the ozone density test. Ozone density around the positive polarity emitter was much lower than that around the negative polarity emitter. These anomalous results are explained as follows. The ozone molecule and the oxygen molecule tend to be negatively charged and act as oxidizers ¹¹⁾. These molecules are attracted to the positive polarity emitter on which they form an oxide film.

5. Conclusion

The particle generation characteristics and the deterioration characteristics were investigated for tungsten and silicon emitters operated in air, nitrogen, and argon atmospheres to determine the most suitable emitter material and gas for the jet ionizer. The experimental results are summarized as follows.

- (1) The number of particles generated from the silicon emitter is less than that generated from the tungsten emitter in each type of atmosphere.
- (2) The number of particles generated in nitrogen atmosphere or argon atmosphere is much lower than that generated in air atmosphere for each type of emitter.
- (3) The number of particles generated from the silicon emitter operated in nitrogen or argon atmosphere is the lowest and is about 200 times smaller than that generated from the tungsten emitter operated in air.
- (4) A silicon emitter operated in nitrogen or argon atmosphere is the most suitable for reducing the number of generated particles.

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