Visualization and Control of Particulate Contamination Phenomena in a Plasma Enhanced CVD Reactor

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Abstract

Dust particle generation in a plasma enhanced CVD (PECVD) process and substrate contamination with the generated dust were studied. By using a laser light scattering technique, the spatial distribution of dust particles suspended in a PECVD reactor was visualized during the fabrication of silicon dioxide thin films. The dust particle size and film morphology were also measured with a laser particle counter and a scanning electron microscope. It was found that the operating conditions of the reactor influenced strongly the properties and transport of dust particles and thus substrate contamination. The effect of the modulation of the plasma input power was also investigated. A new modulation method showed a good performance in terms of both dust suppression and film growth rate.

I. Introduction

Plasma-enhanced chemical vapor deposition (PECVD) of silicon dioxide thin films using a tetraethylorthosilicate (TEOS) and oxygen plasma has become a viable technique in a very large scale integration (VLSI) technology (Tochitani et al. 1993). Silicon dioxide films prepared by this method have demonstrated good step coverage (Wickramanayaka et al. 1997), as well as electrical (DeCrosta and Hackenberg 1996) and mechanical properties (Ramkumar et al. 1993) preferable for use in interlayers, gate oxides, and passivation layers in VLSI technology. However, it has been shown that dust particles tend to generate during the film deposition that leads to the degradation of the

film surface morphology (Fujimoto et al. 2000).

Dust particles generated in the plasma become negatively charged due to the higher mobility of electrons over ions and consequently undergo levitation by the large electric field in the sheath region. Several methods for particulate contamination control in the plasma processes have been proposed that include the use of gas flow (Selwyn and Patterson 1992), thermophoresis (Setyawan et al. 2003), and pulse-wave modulation of rf power (Watanabe et al. 1990). The gas flow is used to sweep the particles away from the trap region near the wafer. The thermophoretic force due to temperature gradient repels the trapped particles away from depositing to the wafer, and thus avoiding contamination. Pulse-wave modulation plasma controls particle generation by interrupting the nucleation and growth of particles and it also serves to reduce the confinement of particles in the traps.

In this paper, dust particle generation and particulate contamination control on the substrate in a PECVD process was studied. The performance of new type modulation waveform in the form of sine wave to control particle formation is presented.

II. Experimental works

The system used for the measurements is shown in Fig. 1. The reactor is a parallel plate type radiofrequency (rf) capacitively PECVD reactor (Setyawan et al. 2002). It consists of two cylindrical plates 200 mm in diameter separated by 35 mm. The upper plate, which is in showerhead configuration, is coupled to a 13.56 MHz rf power supply and the lower plate is grounded. The lower plate is equipped with an electrical heater with an automatic temperature controller. We also performed experiments by modulating the rf power in the form of sine wave. This new type of waveform was aimed to improve the performance of the previously developed waveform, i.e. pulse-wave, which lacks of stability during its operation.



Fig. 1. Experimental setup and the arrangement of the particle measuring system

A mixture of TEOS diluted in nitrogen gas as the carrier and oxygen with a controlled flow rate and composition was introduced into the plasma reactor through the showerhead. Particle formation was observed by means of a laser light scattering (LLS) technique. In this case, a laser beam derived from an Ar⁺ laser (model 2017, Spectra Physics) was expanded into a sheet by a rod and cylindrical lens to illuminate the interelectrodes space through one of the window in the reactor. The light scattered by the particles was detected by a CCD camera positioned perpendicular to the light sheet.

The particle size and the film morphology were measured by a laser particle counter and a scanning electron microscopy (SEM), respectively. To measure the particle size, the particles were drawn from the trap location by inserting a sampling tube into the plasma reactor. The drawn particles were passed through a measuring cell and the number was counted by a laser scattering technique.

III. Results and Discussion

Fig. 2 shows video images of particle clouds below the showerhead at gas flow rates of 60 and 200 sccm. The experiments were carried out at a pressure of 4 Torr, TEOS concentration 5.0%, and ambient temperature. The particle clouds are located in discrete, localized regions between the showerhead holes. The trap regions are static points where particles are not affected so much by the gas drag force. The generated particles form a lump cloud when a low gas flow rate is used, change to a line shape when the flow rate is increased, and eventually the LLS technique can no longer detect them when high gas rate exceeds 400 sccm. When the gas flow rate is high, the neutral drag force presses the clouds more strongly without changing significantly the equilibrium position of the clouds that causes the clouds to shrink.





Fig. 3 shows the effect of gas flow rate on particle formation, represented by the intensity of the scattered light, at room temperature. Other conditions are the same as before. The scattered light intensity decreases when the gas flow rate is increased. It appears that particle formation is suppressed at high gas flow rate. The residence times of TEOS vapor, intermediates, and primary particles in the plasma reactor become longer when the gas flow rate is decreased. This provides more time for the primary particles formed to grow, resulting in a larger particle size. At high gas flow rate, the residence time of the process gases is low and because of this a high gas flow rate tends to remove potential particle generation from the sheath before they are able to form

particles in the nucleation sites and provides less opportunity for the primary particles to grow. SEM photographs of the particles (not shown) confirm that the particles are larger at a low gas flow rate and are smaller at a high gas flow rate.



Fig. 3. Effect of gas flow rate on the intensity of the scattered light (P = 100 Pa, rf power = 100 W, TEOS concentration = 5.0%, O₂:N₂ = 1:1).

In order to clarify the effect of particle generation on particle contamination on the wafer, particles deposited on the silicon wafer during silicon dioxide thin film deposition were observed using SEM. The substrate temperature was set at 300°C with other conditions were the same as before. Figs. 4a-c shows SEM images of particles deposited on the film for gas flow rates of 100 sccm, 400 scm, and 1000 sccm, respectively. It appears that particle contamination decreases with increasing gas flow rate.



Fig. 4. SEM images of particles deposited on the wafer at various gas flow rates: (a) 100 sccm, (b) 400 sccm, and (c) 1000 sccm.

We also observed the cross sectional view of the film using SEM in order to examine the effect of gas flow rate on the film quality. The experimental conditions are the same as before. Fig. 5 shows the SEM images of the cross sectional view of the films at gas flow rates of 100 sccm (a) and 1000 sccm (b). At a gas flow rate of 100 sccm, no particles can be observed to be embedded into the growing film. On the other hand, at a gas flow rate of 1000 sccm, particles are observed to be embedded into the growing film. This suggests that the particles are incorporated into the film during the deposition process, not at the end of the process when the plasma is turned off. In order to control particle generation and growth in the plasma reactor, a new type of waveform modulation plasma, i.e. sine-wave modulation, was developed since the existing modulation plasma, i.e. pulse-wave modulation, has been shown to lack of stability and reproducibility. Fig. 6 shows the size of particles generated in the plasma rector for different modulation waveform. We can see that when the plasma is modulated with sine wave, the particle size is reduced over a broad range of modulation frequency. Even though the pulse-wave modulation also shows reduction in particle size, but it is limited only in the range of low modulation frequencies. When the modulation frequency is higher than 1000 MHz, the particle size is nearly the same as that of continuous wave. It is clearly shown that the sine-wave modulation plasma has a better performance in term of the suppression of particle formation compared to its counterpart pulse-wave modulation.



Fig. 5. SEM images of the cross sectional view of the silicon dioxide film at gas flow rates: (a) 100 sccm, and (b) 1000 sccm.



Fig. 6. The average size of particles generated in the plasma reactor for different modulation waveforms.

Fig. 7 shows the film growth rate for different modulation waveform. We can see that the film growth rate using sine-wave modulation does not show any significant change compared to that of continuous wave over all range of modulation frequency used. This differs from the case of pulse-wave modulation in which the film growth rate is reduced at low modulation frequency, the range where the particle formation is reduced. Thus, the sine-wave modulation also shows better performance in term of film growth rate compared to its counterpart pulse-wave modulation.



Fig. 7. Effect of modulation waveform on the film growth rate.

IV. Conclusion

It has been demonstrated that the plasma operating conditions strongly influences particle formation and transport and thus substrate contamination. Particle formation is suppressed with increasing gas flow rate and temperature, and is enhanced with increasing rf power. The new developed modulation waveform, i.e. sine-wave modulation, has shown a better performance in terms of dust particle suppression and film growth rate than those of its counterpart pulse-wave modulation.

References

- DeCrosta, D. A., and Hackenberg, J. J. (1996). Charge Issues in High Oxygen Gas Ratio Tetraethylorthosilicate Plasma Enhanced Chemical Vapor Deposition Films, *J. Vac. Sci. Technol. A* 14:709-713.
- Fujimoto, T., Okuyama, K., Shimada, M., Fujishige, Y., Adachi, M., and Matsui, I. (2000). Particle Generation and Thin Film Surface Morphology in the Tetraethylorthosilicate/ Oxygen Plasma Enhanced Chemical Vapor Deposition Process, *J. Appl. Phys.* 88:3047-3052.
- Ramkumar, K., Gosh, S. K., and Saxena, A. N. (1993). Stress Variations in TEOS-Based SiO₂ Films During Ex-Situ Thermal Cycling, *J. Electrochem. Soc.* 140:2669-2672.
- Selwyn, G. S., and Patterson, E. F. (1992). Plasma Particulate Contamination Control. II. Self-Cleaning Tool Design, *J. Vac. Sci. Technol A* 10:1053-1059.
- Setyawan, H., Shimada, M., and Okuyama, K. (2002). Characterization of Fine Particle Trapping in a Plasma-Enhanced Chemical Vapor Deposition Reactor, *J. Appl. Phys.* 92:5525-5531.
- Setyawan, H., Shimada, M., Imajo, Y., Hayashi, Y., and Okuyama, K. (2003). Characterization of Particle Contamination in Process Steps during Plasma-Enhanced Chemical Vapor Deposition Operation, *J. Aerosol Sci.* 34:923-936.

- Setyawan, H., Shimada, M., Hayashi, Y., Okuyama, K., and Yokoyama, S. (2004). Particle Formation and Trapping Behavior in a TEOS/O₂ Plasma and Their Effects on Contamination of a Si Wafer, *Aerosol Sci. Technol.* 38:120-127.
- Tochitani, G., Shimozuma, M., and Tagashira, H. (1993). Deposition of Silicon Oxide Films from TEOS by Low Frequency plasma Chemical Vapor Deposition, *J. Vac. Sci. Technol. A* 11:400-405.
- Watanabe, Y., Shiratani, M., and Makino, H. (1990). Powder-Free Plasma Chemical Vapor Deposition of Hydrogenated Amorphous Silicon With High rf Power Density Using Modulated rf Discharge, *Appl. Phys. Lett.* 57:1616-1618.
- Wickramanayaka, S., Nakanishi, Y., and Hatanaka, Y. (1997). On the Chemistry of a-SiO₂ Deposition by Plasma Enhanced CVD, *Appl. Surface Sci.* 113:670-674.