Removal of particles during plasma processes using a collector based on the properties of particles suspended in the plasma

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(Received 9 September 2004; accepted 18 January 2005; published 21 April 2005)

A particle removal system based on the properties of charged particles suspended in a plasma for use in controlling particle contamination during the preparation of silicon dioxide thin films in a plasma-enhanced chemical vapor deposition reactor is described. Since the particles suspended in the plasma carry a negative charge, the application of a positive bias to a metal tube inserted into the plasma would attract negatively charged particles. The system effectively removes particles from the trap regions during operation of the plasma. Even particles as small as about 10 nm in size can be removed using this method. Films prepared using the installed particle removal system were found to be nearly free from particle contamination. This is different from the case when the particle removal system is not installed, where some particles are deposited on the film. Even though the particle removal system reduces the rate of film growth by about 40%, it is offset by the resulting clean film, which is free from particle contamination. © 2005 American Vacuum Society. [DOI: 10.1116/1.1874134]

I. INTRODUCTION

The generation of dust particles is associated with practically all plasma-assisted processes that are currently used in semiconductor manufacturing, including etching,1 deposition,2 and sputtering.3 The presence of dust particles in a plasma reactor may lead to wafer contamination that can seriously degrade the quality of the resulting products. It has been shown that particle contamination may occur under continuous plasma operation or at the end of the process when the plasma is turned off, a factor that depends on the plasma operating conditions employed.4,5 The complete elimination of particle generation during plasma processes would be highly desirable. However, it is unlikely that this can be accomplished because in some cases it is inherent to the plasma chemistry.6,7 Hence, the best strategy for controlling contamination is to limit the generation of particles by plasma chemistry, and to simultaneously influence particle transport in such a way that the generated particles are not deposited on the substrate.

Several methods for controlling particle contamination in plasma processes have been proposed including the use of a gas flow,8 thermophoresis,4,9 and pulse-wave modulation plasma.10 The drag force due to the gas flow sweeps trapped particles away from the location near the wafer, usually into the pump port. A high gas flow rate is also effective in suppressing particle growth.5,11 However, it has been shown that a high gas flow rate tends to cause particles that are trapped at the plasma-sheath boundary near the substrate to escape from the plasma, thus contributing to substrate contamination.11 A thermophoretic force due to a temperature gradient repels the trapped particles away from the surface of the wafer, and thus avoids contamination. To accomplish this, the temperature of both electrodes should be controlled to obtain a desired level of temperature gradient in the gas between the electrodes to ensure the effectiveness of this method. The temperature level is sometimes so high that the film cannot be produced safely. A pulse-wave modulation plasma interrupts the nucleation and growth of particles and reduces the confinement of particles in the traps. A combination of pulse-wave modulation and a gas temperature gradient has been shown to be effective in reducing the particle growth.12 However, the application of pulse-wave modulation to a rf plasma leads to an unstable operation and may alter the process conditions.13

In the present work, we report on the use of a negatively-charged fine particle (NFP) collector, developed by Sato et al.,14 to control particle contamination in a capacitively coupled rf plasma-enhanced chemical vapor deposition (PECVD) reactor. The NFP collector consists of a metal tube with an applied positive bias that is inserted into the plasma space. The particles suspended in the plasma are drawn into the tube solely by the developed electric field near the collector surface. The method was developed based on the fact that most particles suspended in the plasma are negatively charged.15 Hence, by applying a positive bias to a metal tube inserted into the plasma, negatively charged particles would be attracted by the electrostatic force into the tube. This method has been successfully applied to the removal of fine particles in a remote magnetron-type rf plasma, which is divided into regions of plasma generation and deposition, for
the deposition of $\alpha$-Si:H films. Thus, the deposition was carried out in the afterglow plasma. The particle collectors were installed in the deposition region, arranged on metal blocks with their faces up and their top surfaces level with the substrate. The same design as was in the remote magnetron-type rf plasma might not be applied directly to a widely used parallel-plate type rf plasma reactor because of the differences in reactor configuration and the method of plasma generation. In the case of the widely used parallel-plate type rf plasma, the regions of plasma generation and deposition are not separated and the plasma is solely generated by a rf power without any assistance of a magnetic field. Consequently, the characteristics of the particle collector may also be different. Therefore, it is necessary to develop a new design for a particle collector that matches the reactor configuration and to understand the process conditions required for a parallel-plate type rf plasma reactor.

In this article, we report on the performance of a newly designed particle collector in controlling particle contamination during the preparation of silicon dioxide thin films using a tetraethylorthosilicate (TEOS) and oxygen. The basic characteristics of the particle collector are discussed based on the observation of particles collected by the collector and the visualization of particle motion around the collector. The effects of the particle collector on wafer contamination and film growth rate are presented.

II. EXPERIMENTAL WORKS

Experiments were carried out in a parallel-plate type, 13.56 MHz rf capacitively coupled PECVD reactor. The reactor used has been described in detail elsewhere. Briefly, it consists of two cylindrical plates 200 mm in diameter separated by 35 mm. The upper plate, which is in a showerhead configuration, is coupled to the rf power and the lower plate is grounded. The lower plate is equipped with an electrical heater with an automatic temperature controller.

Two types of particle collectors were employed in this study, namely a tube-type and a ring-type, as shown in Figs. 1(a) and 1(b), respectively. The tube-type particle collector consists of a 4.0 mm i.d. stainless steel tube insulated with a silicone tube except for the tip. Thus, only the tip of the stainless steel tube is exposed to the plasma. The tube was connected to a dc voltage regulator to permit the bias voltage to be adjusted. The tube was installed in the reactor at a location about 3 mm below the showerhead where most of the generated particles are trapped. This arrangement was used to investigate the basic characteristics of the particle collector. The particles can be either injected from the outside in a chemically inert plasma or generated by gas phase nucleation using a TEOS/O$_2$ plasma for the preparation of silicon dioxide thin films. The behavior of the particles around the particle collector was observed using a laser light scattering (LLS) technique combined with video imaging. A laser beam derived from an Ar$^+$ laser (Model 2017, Spectra Physics) was expanded to form a light sheet parallel to the showerhead surface using a rod lens in order to illuminate the region near the particle collector. A high resolution charge coupled device (CCD) video camera equipped with an interference filter was used to record particle motion. The configuration of the laser light and the video camera is shown in Fig. 1(a). The particles collected by the particle collector were deposited on a chopped Si wafer of 2.0 mm $\times$ 2.0 mm in size placed about 5 mm from the tip of the tube and then observed by scanning electron microscopy (SEM).

The ring-type particle collector consists of a ring 220 mm in inner diameter, electrically insulated by an insulator material, in which 32 stainless tubes 4.0 mm in inner diameter connected to a dc voltage regulator are flush mounted with the inner ring surface. The stainless steel tubes serve as the particle collector, analogous to the tube-type particle collector. The inner surface of the ring is insulated to minimize the effect of the particle collector on plasma properties. When an insulator is immersed in a plasma, it will be at the floating potential, i.e., no net current to the surface, and thus, the disturbance caused by the presence of the particle collector is localized. The ring was installed such that the position of the holes was 3.0 mm below the showerhead, about at the level of the trapped particle position. The ring-type particle collector was designed based on our previous observations of the behavior of particles trapped in our system. Our previous findings indicated that when the trapped particle concentration at a given plasma condition has reached saturation, the particles are released from the trap through the periphery of the showerhead. Using this arrangement, the removal of particles from the trap location through the periphery of the...
showerhead would be expected to be accelerated and effective. A chopped wafer was also placed in one of the holes to observe the collected particles.

In order to evaluate the performance of the ring-type particle collector in controlling particle contamination, it was installed in a PECVD reactor that was used for the preparation of silicon dioxide thin films using TEOS/O₂ gases with nitrogen as the carrier for the TEOS vapor. All evaluations of the performance of the ring-type particle collector were carried out at fixed plasma conditions. The total gas flow rate and TEOS concentration were 100 sccm and 5.0%, respectively, and the ratio of nitrogen to oxygen was 1:1. The deposition was conducted at a pressure of 3.0 Torr and a substrate temperature of 300 °C. The particles collected by the ring-type particle collector and deposited on the wafer placed on the grounded electrode were observed by SEM. The rate of film growth was measured by means of a surface texture-measuring instrument (Surfcom 1400D, Accretech, Japan).

III. RESULTS AND DISCUSSION

A. Basic characteristics of particle collector

In order to design and operate the particle collector appropriately, it is necessary to understand the basic characteristics of the particle collector in relation to plasma operating conditions. It is desirable that the particle collector be able to remove a broad range of particle sizes because particles generated in the plasma processes may range from a few nanometers to several micrometers in size. Since this method works based on the electric field created by positively biasing the particle collector so as to attract negatively charged particles, the performance of the collector is dependent on the applied bias voltage. The electric field formed in the space surrounding the tubes attracts charged particles via electrostatic forces. The electrostatic force is proportional to the electric field intensity and the particle charge, i.e., the particle size. Consequently, the particle collector performance may be influenced by the applied bias voltage from the stand point of the particle size to be removed. Hence, in an initial step experiment, we attempted to determine the range of bias voltage required to remove a broad range of particle sizes. For this purpose, we used the experimental setup shown in Fig. 1(a).

In order to determine the particle size that can be removed by the particle collector, an Si wafer was placed inside the tube and the particles deposited on it were observed by SEM. These particles can be regarded as particles collected by the particle collector. Figure 2 shows SEM images of particles deposited on the Si wafer at various bias voltages by seeding the plasma with a mixture of monodisperse silica particles with sizes 0.6 and 2.5 μm. In this case, the particles were injected into the reactor from the outside through the showerhead. The particle injection system has been described elsewhere. The plasma was derived from nitrogen gas at a pressure of 3.0 Torr and room temperature. It can be seen that both sizes were removed from the plasma at a bias voltage of +40 V. On the other hand, only 2.5 μm-size particles were removed at bias voltages of +20 and +80 V. Thus, the applied bias voltage strongly influences the size of the particle that can be removed from the plasma. A qualitative explanation for this may be given as follows. When a bias voltage is applied to the particle collector, several behaviors may be observed that depend on the relative value of the bias voltage with respect to the plasma potential rather than its absolute value. Three conditions may be observed: (i) the particle collector is at the same potential as the plasma, (ii) the particle collector is positive relative to the plasma potential, and (iii) the particle collector is negative relative to the plasma potential. Each condition and its effect on particle removal will be discussed below.

If the collector is at the same potential as the plasma, no electric field is generated around the collector. At this condition, it is assumed that no particles are removed by the particle collector. If the particle collector is positive relative to the plasma potential, an electric field will be generated in front of the particle collector with the gradient directed away from the collector. At this condition, negatively charged particles are attracted to the collector as the result of an electrostatic force. Near the collector surface, an excess of negative charge is produced as the electrons are attracted to the positive body. This negative charge will act as a repulsive barrier for the negatively charged particles to reach the collector. If the kinetic energy of the particle due to the attractive electrostatic force is not sufficient to overcome this repulsive barrier, the particle cannot pass through the sheath. As a result, it will not be collected. The excess negative charge near the collector surface increases with increasing bias voltage. Moreover, the electrostatic force increases with increasing particle size. Thus, when the relative potential between the collector and the plasma is relatively low, only a few electrons accumulate in front of the particle collector voltage. Consequently, the repulsive barrier becomes weak, which permits small particles that have a lower kinetic energy due to the attractive electrostatic force to overcome the potential barrier. They then pass through the barrier, and are collected by the particle collector. This may explain why, at a bias voltage of +40 V, i.e., a low relative potential, both sizes are removed and at a bias voltage of +80 V, i.e., a relatively high potential, only large particles are removed.

If the particle collector is negative relative to the plasma potential, electric fields will also be generated, but with the

![Fig. 2. Effect of bias voltage on the particle size that can be removed by the tube-type particle collector: (a) +20 V, (b) +40 V, and (c) +80 V. Silica particles, 0.6- and 2.5-μm in size, are injected into the reactor from the outside.](image-url)
gradient directed to the collector. Instead of attracting electrons, the particle collector attracts positive ions so that it will be surrounded by an ion sheath layer. Ions flowing into the collector will drag the particle toward the collector surface. This ion drag force is proportional to the square of the particle diameter. Thus, the particle kinetic energy due to this ion drag force increases with increasing particle size. This may explain why large particles are able to overcome the repulsive barrier of the negatively bias collector and are removed, whereas the small ones are not.

In order to better understand how the particles are removed by the particle collector, we visualized the particle motion around the collector using the LLS technique described above. Figure 3 shows the time variation of video images near the tube-type particle collector after turning it to a bias voltage +40 V, using 2.5 μm-size particles. The plasma operating conditions were the same as those for monodisperse particles injected from the outside. The results clearly show that particles are attracted to the particle collector. Only 17 s are required to remove the particles after turning on the particle collector. This is much shorter than the time required for particles to be visualized during a silicon dioxide thin film preparation using TEOS/O₂, which typically requires about 2 min. This indicates that the rate of removal of particles is much higher than the rate of particle formation. Thus, the particle collector is effective even in a system with continuous particle formation.

Figure 4 shows video images comparing the conditions of the trapped particles generated in TEOS/O₂ plasma without any particle collector (a) and in the presence of a particle collector at a bias voltage of +40 V (b). In this case, the particles are generated by gas phase nucleation, followed by particle growth through coagulation and/or agglomeration. The experiments were carried out at a pressure of 3.0 Torr and room temperature. The total gas flow rate and TEOS concentration were 50 sccm and 1.0%, respectively, with a ratio of nitrogen to oxygen of 1:1. It can clearly be seen that without the particle collector, numerous particle clouds are formed below the showerhead. On the other hand, in the presence of the particle collector, the particle clouds have nearly disappeared. This shows that the particle collector is very effective in removing generated particles during the deposition process in a plasma reactor.

B. Contamination control using ring-type particle collector

In order to investigate the effectiveness of this method for controlling particle contamination in a plasma reactor, the ring-type particle collector was installed in the PECVD reactor for the preparation of silicon dioxide thin films using a TEOS/O₂ plasma. Since the position of the particle collector and the plasma operating conditions are slightly different from those for the tube-type particle collector, we again varied the applied bias voltage of the particle collector around the best value obtained for the tube-type particle collector. In the tube-type particle collector, the collector is inserted deep into the plasma whereas in the case of ring-type particle collector, it is located just outside the plasma generation space.

As in the case of the tube-type particle collector, a Si wafer was placed inside the collecting holes of the ring-type particle collector and the particles deposited on it were observed by SEM. Figure 5 shows SEM images of particles deposited on the Si wafer at various bias voltages. At bias voltages of +40 V and +60 V, the collected particles were in the range from a few nanometers to more than 10 μm. Figure 6 shows the size distribution of particles collected by the ring-type particle collector at a bias voltage of +40 V. The distribution is very broad with the peak located in the range size of 0–100 nm. This shows that the particle collector is very effective for removing particles from the plasma space.

Figure 7 shows SEM images of the prepared thin film at conditions of (a) without operating the ring-type particle col-
lector, and (b) operating the ring-type particle collector at a bias voltage of +40 V. The plasma operating conditions were the same for the two cases. It can be seen that some particles contaminate the wafer placed on the grounded electrode when the ring-type particle collector is not in operation. This is different from the case where the ring-type particle collector is operated. In this case, the wafer is almost free from particle contamination. This clearly shows that the ring-type particle collector effectively removes particles from the plasma space.

C. Effect of the ring-type particle collector on film growth rate

Figure 8 shows the effect of applied bias voltage on film deposition rate for silicon dioxide using TEOS/O₂. The plasma operating condition is the same as that used in the previous experiments. While particle contamination on the film is greatly reduced by the ring-type particle collector, the film growth rate also tends to decrease. Thus, a trade-off exists between particle contamination on the film and the film growth rate when the ring-type particle collector is operated. The particle collector reduces the film growth rate by about 40%. This reduction probably is consistent with the mechanism of silicon dioxide deposition. It has been shown that the growth rate of silicon dioxide film using a TEOS/O₂ plasma depends on the sum of the oxygen atom induced and the ion-assisted pathways.20 By applying a bias voltage to the particle collector that is located near the power electrode, some ions responsible for the film deposition may be trapped.
in this region. Hence, the concentration of these ions near the wafer decreases, which causes a decrease in film deposition rate due to the lower contribution of ion-induced deposition.

We investigated the effects of the applied bias voltage on the film thickness uniformity by measuring the thickness profile across the wafer using a surface texture-measuring instrument (Surfcom 1400D, Accretech, Japan). The plasma operating conditions were the same as those in the previous experiments. It was found that the uniformity was virtually the same for all bias voltages. Observation of the films using an atomic force microscope (AFM) also indicated that a film of a good surface smoothness (RMS roughness of about 0.2 nm) was obtained at all bias voltages.

IV. CONCLUSION

It has been demonstrated that a particle collector with a positive bias voltage effectively removes dust particles generated in the plasma process for a silicon dioxide thin film preparation using TEOS/O₂ gases. Particle sizes ranging from a few nanometers to several micrometers are successfully removed from the plasma space. This confirms that it is possible to control particle contamination during film deposition. The film deposited on a wafer placed on the grounded electrode is free from particle contamination when the particle collector is in operation. A trade-off exists between particle contamination on the film and the film growth rate when the ring-type particle collector is used. While the ring-type particle collector greatly reduces particle contamination on the film, it also slightly reduces the film growth rate. The reduction in film growth rate is offset by the production of a clean film that is nearly free from particle contamination.

ACKNOWLEDGMENTS

This work was partly supported by innovation Plaza Hiroshima of JST (Japan Science and Technology Agency) and a Grant-in-Aid from the Ministry of Education, Sports, Culture, Science, and Technology of Japan.