SURFACE MORPHOLOGY MEASUREMENTS AND PARTICLE FORMATION ON SiO_x FILMS DEPOSITED USING LOW FREQUENCY PLASMA CVD METHOD

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Abstract

The surface morphology of SiO_x films and, in particular, dust particle formation dynamics on those films deposited from a SiH_4 and N_2O mixture using a low frequency plasma enhanced chemical vapor deposition (PECVD) have been investigated by scanning electron microscopy (SEM), and by SEM and laser light scattering (LLS), respectively. With respect to surface morphology, the morphology size D of the films deposited without substrate heating is found to vary with the film thickness t as $D=t^x$ where x=0.67 on both Si and glass, with both discharge frequencies f=50Hz and 1kHz. It is observed by the SEM that particles are deposited above f=5kHz both with and without substrate heating (400℃), while no particles are deposited below f=1kHz. Moreover, LLS indicates that particles are generated above f=3kHz in the gas phase, and that they are not generated in the gas phase below f=3kHz. Films with particles have properties (the refractive index, resistivity and Vickers hardness) inferior to films without particles. In this paper, we show experimentally that particle-free nature of the low frequency PECVD method (less than f=3kHz) results in high quality films without requiring of substrate heating.

1. Introduction

Recently, silicon oxide films have been utilized for passivation and as buffer layer in integrated circuits', therefore understanding their morphology is important to improve reliability of semiconductor devices. Despite its importance, little has been reported about the morphology of silicon oxide film surfaces. Here we present the first report of the dependence of the surface morphology on the film thickness of silicon oxide films deposited by a low frequency PECVD method using a SiH_4 and N_2O mixture'.

It has generally been accepted that good films can not be obtained using RF-PECVD methods without substrate heating because of particle generation [1]. Nonetheless, it has been reported by the authors' that transparent silicon oxide films having good properties can be obtained by a 50Hz PECVD method without substrate heating [2]. Therefore it seems that particle generation depends on the discharge frequency. Next, the effect of the discharge frequency, f, on the particle formation and on properties such as the refractive index, resistivity and Vickers hardness of the silicon oxide films are investigated. Finally, from the viewpoint of particle generation, it is discussed why film having good properties can be deposited using a low frequency PECVD method even without substrate heating.

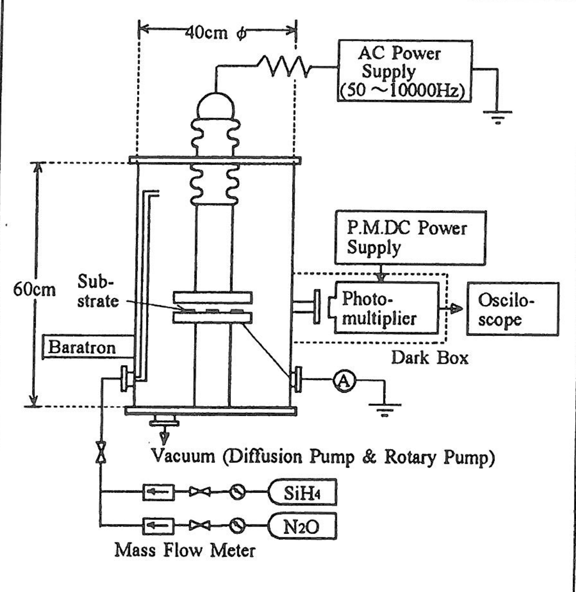


Fig. 1 Reactor

2. Experimental

Fig. 1 shows the deposition reactor used in this experiment. The stainless steel reaction chamber is equipped with the parallel plane electrodes (ϕ =15cm, gap length 2cm). SiH_4 and N_2O used in this experiment are of high purity (99.999%). Substrates on the grounded electrode are cut into $1cm^2$ squares. Fig. 2 shows the position of the LLS [3] measurements in the chamber. A particle cloud is assumed to be present 6mm above the lower electrode based on results from a preliminary experiment. The discharge conditions for the film deposition and for LLS are shown in Table 1. The deposition conditions relating to sections 3.1 and 3.2 are shown in Table 1 (A) and (B), respectively. Elementary analysis of the films deposited for the condition shown in Table 1 (A) was performed by means of Auger electron spectroscopy and X-ray photo-electron spectroscopy. The films are found to consist of only Si and O.

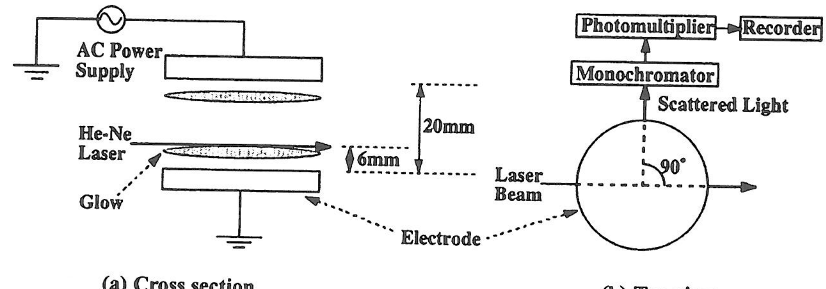
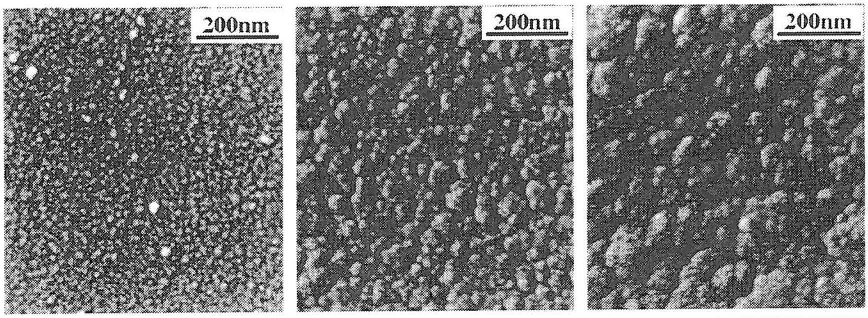


Fig. 2 Position of the LLS measurements



(a) (b) (c)

Fig. 3 Scanning electron micro-graphs of silicon oxide films deposited on Si at *f*=50Hz without substrate heating. Film thickness is 40nm (a), 650nm (b) and 870nm (c).

3. Results and Discussion

3.1 Morphological Evolution of Silicon Oxide Film Surface with Increasing Film Thickness

As shown in Fig. 3, the diameter of circular protuberances on the surface of the films deposited on Si without substrate heating at *f*=50Hz increases gradually with film thickness, while the cross section of the films shown in Fig. 4 indicates that the films are not porous. The morphology size is defined using feature diameters to introduce a quantitative parameter which describes morphological roughness. The morphology size is averaged from five measurements. As shown in Fig. 5, the films deposited without substrate heating for both Si and glass substrates and frequencies from *f*=50Hz to 1kHz seem to have the same morphology as shown in Fig. 3. Fig. 6 and 7 show a comparison of the evolution of the morphology size with increasing thickness of the films deposited without substrate heating on Si and on glass, and at *f*=50Hz with at *f*=1kHz, respectively. In these figures, there is no morphological difference between those films on Si and glass, and between those at *f*=50Hz and 1kHz. As shown in Fig. 8, the films deposited below *f*=1kHz at 400℃ are very smooth. This suggests the following mechanism. Key radicals impinge on the protuberances more intensely than on concave areas, and are fixed there if surface diffusion is slow due to the low substrate temperature or the low kinetic energy of key radicals [4,5]. So, the

Table 1 Discharge Conditions

	Deposition		LLS
	(A)	(B)	
Flow Rate (sccm) ; SiH_4	3.4	3.4	3.4
; N_2O	134	134	134
SiH_4 Mixture Ratio (%)	2.5	2.5	2.5
Pressure (Torr)	0.5	0.5	0.7
Discharge Frequency (Hz)	50～1000	50～10000	～20000
Plasma Current (mA)	4～8	8	8
Substrate	Si, Glass	Si	
Substrate Temperature (℃)	25, 400	25, 400	25

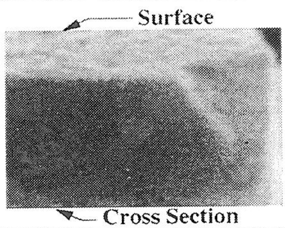
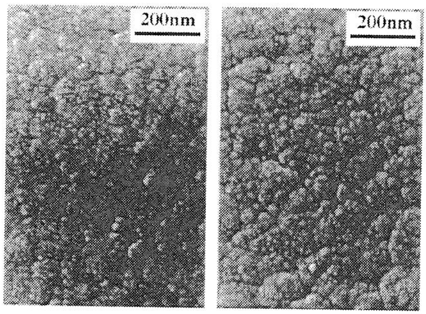


Fig. 4 Cross section of the SiO_x film deposited at *f*=50Hz on Si without substrate heating.

protuberances grow faster than concave regions, and join the next protuberance; as a result, the unevenness is amplified with increasing film thickness. It has been reported that there is the correlation $D = t^x$ between the morphology size D and film thickness t, where x is a constant depending on the experimental conditions [4]. In this paper, we obtained the value of x=0.67 by the method of least squares.



(a) (b)

Fig. 5 Scanning electron micro-graphs of SiOx films deposited without substrate heating ; on glass at f=50Hz (a), on Si at f=1kHz (b).

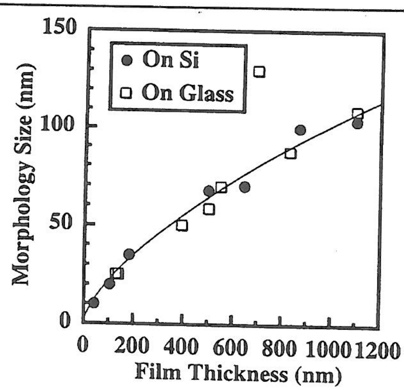


Fig. 6 Morphological evolution of SiOx films versus film thickness; without substrate heating, at f=50Hz.

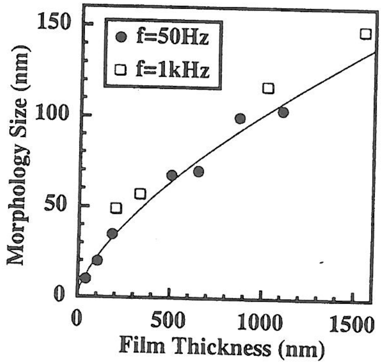
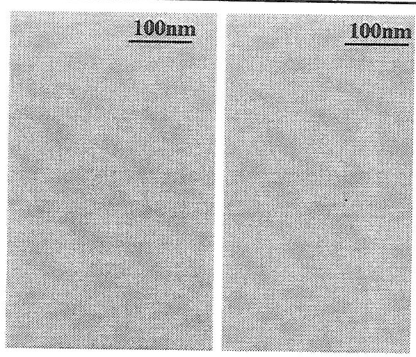


Fig. 7 Morphological evolution of SiOx films versus film thickness; without substrate heating, on Si.



(a) (b)

Fig. 8 Scanning electron micro-graphs of SiOx films deposited at 400℃; (a) f=50Hz, (b) f=1kHz.

3.2 Effect of Discharge Frequency on Particle Formation and Properties of Silicon Oxide Films

Fig. 9 shows SEM photographs of the surfaces of the deposited films at f=10kHz. As seen in Fig. 9 (a) and (b), particles are deposited on the substrates above f=5kHz both with and without heating of the substrate, while no particles are deposited up to f=1kHz as described in section 3.1. The particles on the heated substrates seem to

(a)

(b)

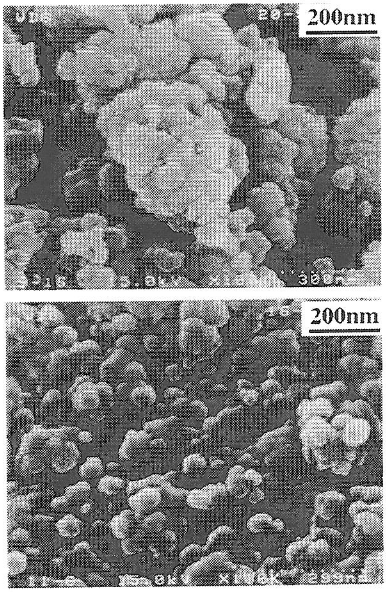


Fig. 9 Scanning electron micro-graphs of SiOx films deposited on glass at *f*=10kHz; without substrate heating (a), at 400℃ (b).

aggregate as seen in the SEM photographs and the films look transparent. On the other hand, the films deposited without substrate heating look white and the particles seem to be disperse. Fig. 10, 11 and 12 show the results of refractive index, Vickers hardness and resistivity measurements, respectively. Values of Vickers hardness and the resistivity of each film with particles decrease considerably, but as far as the refractive index is concerned, the films which consist of tightly aggregated particles deposited with substrate heating do not have values lower than 1.43. From the LLS measurements (Fig. 13), it was found that particles were generated above *f*=3kHz in the gas phase, and that they were not below *f*=3kHz. Watanabe et al. [6] succeeded in suppressing the particle generation by using a square-wave-amplitude-modulated RF discharge as long as the modulating frequency was below f_M=10kHz. Our previous paper pointed out that the time dependence of the emission intensity suggested the discharge is interrupted every halfcycle with low frequency plasmas [7]. The interruption time at *f*=3kHz in this experiment calculated from the emission intensity is very close to that of the modulated RF in Watanabe's experiment (0.05ms). So, we believe that particle precursors such as silanol [8] whose life time is short can not accumulate below *f*=3kHz because of the long interruption time.

4. Conclusions

The morphology size of silicon oxide films deposited from a SiH_4 and N_2O mixture using a low frequency PECVD method without substrate heating was observed using SEM and found to increase with film thickness. The correlation $D=t^x$ between the morphology size D and film thickness t was confirmed with x equal to 0.67. Further, there was no morphological difference found between those films on Si and glass, and between those at *f*=50Hz and lkHz. Dust particles are observed to be generated in the gas phase above *f*=3kHz and not to be generated there below *f*=3kHz. They are deposited both with and without substrate heating (400℃). We suggest that particle-free nature of the low frequency PECVD method less than *f*=3kHz results in high quality films without the need of substrate heating.

Acknowledgments

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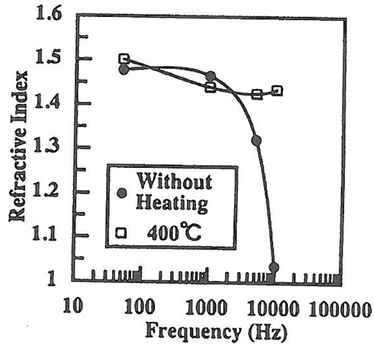

Fig. 10 Refractive index of SiOx films versus frequency

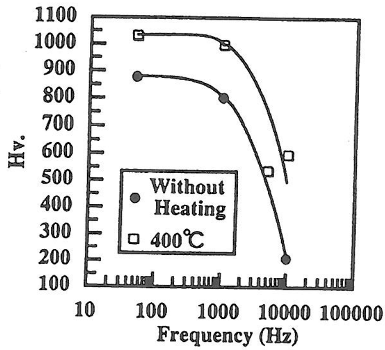

Fig. 11 Vickers hardness Hv. (5gf/15s) of SiOx films versus frequency

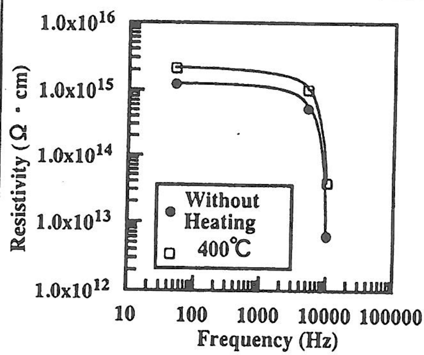

Fig. 12 Resistivity of SiOx films versus frequency

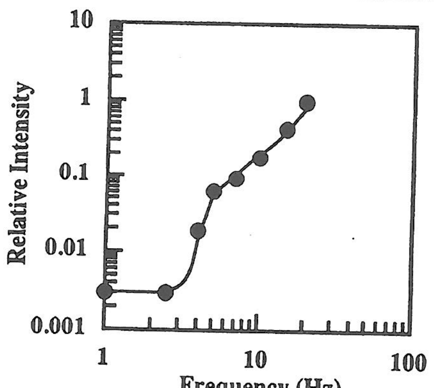

Fig. 13 Relative Intensity of laser light scattered by particles versus frequency