

Comprehensive Investigation on Fluorosilicate Glass Prepared by Temperature-Difference-Based Liquid-Phase Deposition

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This study investigates the properties of fluorosilicate glass film prepared by temperature-difference-based liquid-phase deposition. Experimental results indicate that the deposition temperature can significantly affect the deposition rate and film properties. As the deposition temperature increases from 15 to 35°C, the deposition rate increases exponentially from 30 to 687 Å/h. Meanwhile, the F and OH contents increase from 2.56 to 3.49% and from 0.16 to 0.5%, respectively. The dielectric constants and stresses for the films deposited at 15, 25, and 35°C, are 3.56/63 MPa, 3.46/43 MPa, and 3.66/73 MPa, respectively. The current densities at 2 MV/cm for the films deposited at 15, 25, and 35°C are 6.9×10^{-9} , 4.6×10^{-9} , and 3.6×10^{-7} A/cm², respectively. For the film deposited at 25°C, the dielectric constant and the stress are minimal because of the high F content, low OH content, and low bond strain. The properties are also compared with those of fluorosilicate glass prepared by plasma-enhanced chemical vapor deposition. In addition to exhibiting a lower dielectric constant and much better resistance to moisture, the film prepared by temperature-difference-based liquid-phase deposition also shows a comparable or better insulating property, revealing a reliable intermetal dielectric with a low dielectric constant.

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As device geometry scales down to 0.25 μm and below, the parasitic capacitance between closely spaced metal lines becomes important in terms of the resistance-capacitance time delay for device switching.¹ A novel intermetal dielectric (IMD) with a low dielectric constant (low-*k*) is thus required to reduce the parasitic capacitance. Thus, conventional plasma-enhanced chemical vapor deposited (PECVD) oxide with *k*~4.3 has been gradually replaced by low-*k* (*k*~3.5) fluorosilicate glass (FSG).²⁻⁶ The low-*k* (*k* < 3) spin-on dielectric (SOD) is also a good candidate as a novel IMD. However, the low-*k* SOD generally requires an overlying cap layer to isolate the SOD from moisture and oxygen plasma.⁷⁻⁹ FSG may replace conventional PECVD oxide to cap the SOD for further reducing the parasitic capacitance. In fact, a 10% reduction in parasitic capacitance has been achieved using FSG as a cap layer for a SOD.⁷ As a stand-alone IMD, FSG with a low dielectric constant, low stress, and low deposition temperature is desirable for achieving a good performance and/or reliability.^{1,10,11} Furthermore, as a cap layer for a SOD, there is also a demand for deposition of the FSG without oxygen plasma damage on the underlying SOD.^{8,9} A FSG film prepared by temperature-difference-based liquid-phase deposition (TD-LPD), using a 4 M hydrofluorosilicic acid (H₂SiF₆) solution, has been proposed to satisfy these requirements.¹² Besides, the film with N₂O plasma annealing has been successfully applied as cap layers for low-*k* (*k*~2.8) SODs such as hydrogen silsesquioxane and methylsilsesquioxane.⁹ The dielectric constant and the stress of TD-LPD film are lower than those of conventional LPD film, which requires the addition of a supersaturation initiator to silica-saturated H₂SiF_{6,2}.^{2,12-14} Adding a supersaturation initiator indeed dilutes H₂SiF₆ solution, resulting in less fluorine atoms and more OH bonds incorporated into a conventional LPD film.

Sakai *et al.* first proposed the notion of controlling the degree of supersaturation for the deposition solution by altering the difference between the solution-preparation temperature and the deposition temperature, without adding any supersaturation initiator.¹⁵ Kawahara *et al.* studied the chemical composition, the P-etch rate and the stress for this kind of oxide films deposited at 35 and 60°C.¹⁶ According to their results, a higher solution temperature, *i.e.*, 60°C, and a higher H₂SiF₆ concentration, *i.e.*, 3 M, led to a lower hydroxy content and higher density for the film.

Therefore, using a 4 M H₂SiF₆ solution (without dilution) and the temperature-difference technique proposed by Sakai *et al.*, TD-LPD film is highly promising for future multilevel interconnection

in terms of a low dielectric constant, low stress, high fluorine content, low hydroxy content, and high film density. However, the dependency of film properties on the deposition temperature remains unknown. Therefore, this work investigated the properties for films deposited at 15, 25, and 35°C. The effect of 400°C furnace annealing on the dielectric constant, the stress, and the leakage current were also studied. The properties of TD-LPD film are also compared with those of FSG prepared by PECVD.

Experimental

Figure 1 depicts the flow diagram for TD-LPD process. Silica powder was added to a 4 M H₂SiF₆ solution and then dissolved by stirring at 0°C until the solution became saturated. After removing the undissolved silica by filtering, the saturated solution was heated to 15, 25, or 35°C, and became supersaturated. After cleaning, the wafers were immersed in the solution and, then, the film was gradually deposited onto the wafers. Thus, the differences between the solution-preparation temperature and the three deposition temperatures were 15, 25, or 35°C.

Next, properties of the films prepared at various temperatures were investigated. The thickness and the refractive index were monitored using an ellipsometer. The chemical bonding structure was investigated using Fourier transform infrared spectroscopy (FTIR). For accurate analysis of the peak position and full width at half-maximum (fwhm), for various films, the thickness was controlled to a small range of 3600~4000 Å. The chemical etch rate was evaluated using P-etch solution (HF:HNO₃:H₂O = 3:2:60). The dielectric constant (1 MHz) was then evaluated using a capacitance-voltage (C-V) measurement system with a metal oxide semiconductor (MOS) capacitor structure. The stress of ~2000 Å thick film was calculated from the measured curvature based on Stoney's equation, using a Tencor FLX-2320 instrument. The current density vs. electrical field (*J-E*) characteristic was studied using an HP4145B system also with the MOS structure.

For comparison, a FSG film was prepared using a conventional parallel-plate electrode PECVD reactor operated at a radio frequency of 380 kHz with a power level of 200 W. CF₄ was added as the fluorine source into tetraethylorthosilicate (TEOS)/O₂ at 300°C. The flow rates for CF₄, TEOS, and O₂ were 200, 10, and 400 sccm, respectively. Properties of the PECVD film were also measured or extracted as described above.

Results and Discussion

Results in this study demonstrate that, in using the TD-LPD method, the film can be formed by raising the deposition tempera-

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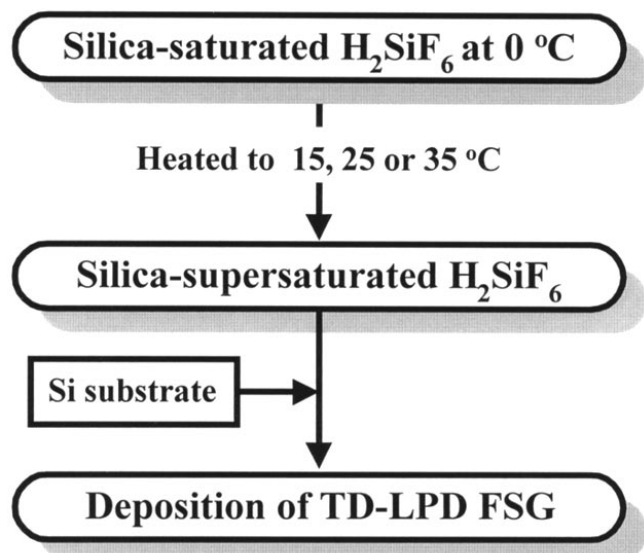


Figure 1. Flow diagram for TD-LPD process.

ture higher than the solution-preparation temperature ($\sim 0^\circ\text{C}$). Besides, the deposition rate depends on the temperature, but not on the deposition time. According to Fig. 2, the rate increases nearly exponentially with an increasing temperature. Therefore, the rate can be controlled well within $30\sim 1200 \text{ \AA/h}$, corresponding to the temperature range of $15\sim 40^\circ\text{C}$. From the relationship between the rate and the temperature, an activation energy of 1.13 eV is obtained. According to a previous investigation, the faster the deposition rate implies a lower likelihood that the atoms reach their lowest energy state and the densest structure. Thus, the resulting films can be quite porous with a significant amount of bond strain in the films.¹⁷ Therefore, as expected, the exponentially increased rate significantly affects the film density and the bond strain.

Figure 3a presents the FTIR spectra for the films deposited at 15, 25, and 35°C , indicating that they resemble each other in this scale. The peaks at approximately 457 , 805 , and 1100 cm^{-1} are attributed to rocking, bending, and stretching vibration for Si-O-Si bonds, respectively. Peaks at 932 cm^{-1} are due to Si-F bonds. The faint peaks around 3680 cm^{-1} are a consequence of Si-OH bonds.¹⁸ These findings reveal that the films are all FSG films with low hydroxy contents.

The peak position and the fwhm associated with Si-O-Si stretching vibration were further investigated and summarized in the inset table in Fig. 3a, where DT denotes the deposition temperature. Ac-

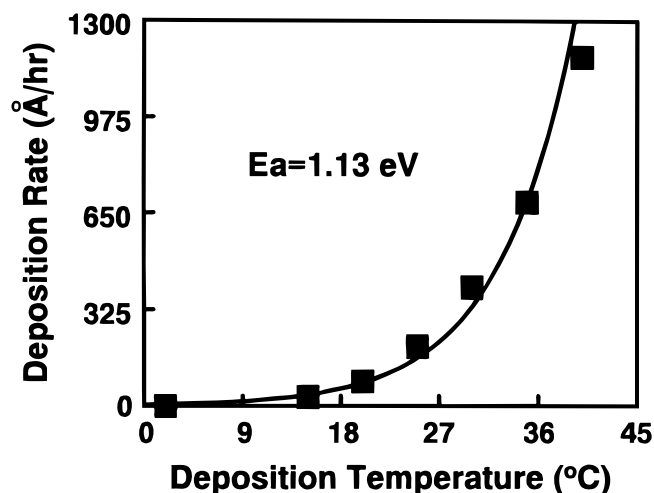
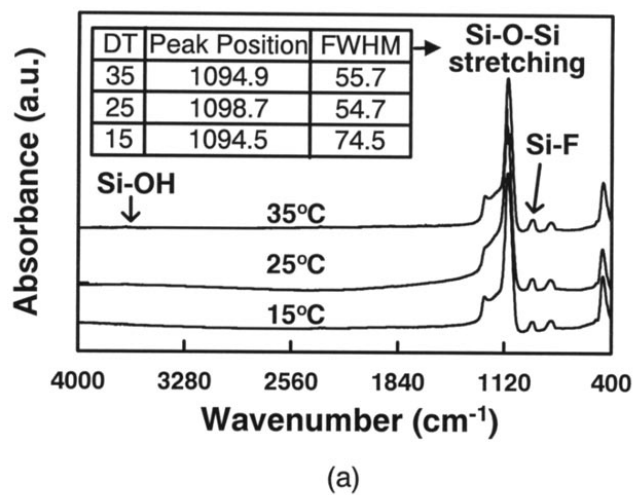


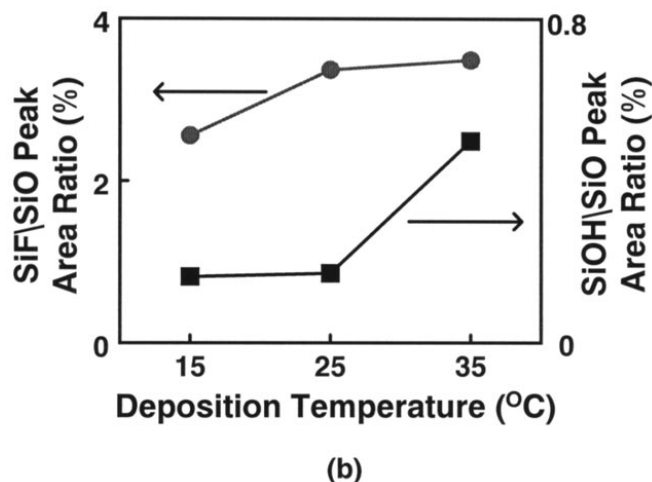
Figure 2. Deposition rate as a function of deposition temperature.

ording to the table, as the temperature increases from 15 to 25°C , the peak position increases from 1094.5 to 1098.7 cm^{-1} ; however, the fwhm decreases from 74.5 to 54.7 cm^{-1} . Nevertheless, as the temperature further increases to 35°C , the peak position decreases to 1094.9 cm^{-1} , while the fwhm slightly increases to 55.7 cm^{-1} . The peak position and the fwhm do not change monotonically with an increasing temperature. This finding implies that two or more factors affect the chemical bonding structure during the temperature changes. In fact, the peak position can shift to a lower wavenumber for the film with more bond strains, leading to a greater fwhm.¹⁷ In contrast, for the film with more fluorine atoms incorporated, the peak position can shift to a higher wavenumber, and the fwhm becomes smaller.¹⁹ Presumably, the bond strain and fluorine atoms in the film dominate the changes in the peak position and the fwhm.

The content for Si-F bond (F content) or Si-OH bond (OH content) is further evaluated by dividing the integrated area for the Si-F peak or the Si-OH peak, respectively, by that for the Si-O-Si stretching peak. According to Fig. 3b, both contents obviously increase with increasing temperature. The F content is 2.56% for the film deposited at 15°C ; it further increases to 3.37 and 3.49% for the films deposited at 25 and 35°C , respectively. This study also measured the F concentration by X-ray photoelectron spectroscopy (XPS)



(a)



(b)

Figure 3. (a) FTIR spectra for films deposited at 15, 25, and 35°C . The inset table shows the position and the fwhm for the Si-O-Si stretching peak. (b) F content and OH content as functions of deposition temperature.

for comparison. The F concentration for the films deposited at 15, 25, and 35°C are 7.0, 8.6, and 8.9 atom %, respectively. The increasing tendency of the F concentration with temperature resembles that of the F content determined by FTIR. Besides, the F concentration for the films prepared by TD-LPD is higher than 1.85~6.2 atom % for the films prepared by conventional LPD.^{14,20,21} Obviously, TD-LPD is an effective means of incorporating fluorine atoms into the films. On the other hand, the OH content is 0.16 or 0.17% for TD-LPD films deposited at 15 or 25°C, respectively. However, the OH content significantly increases to 0.5% for the film deposited at 35°C. The higher the temperature implies that more terminating bonds, *i.e.*, Si-OH and Si-F bonds, are incorporated into the film. Apparently, the F content for 25°C has nearly saturated as compared with that for 35°C; while the OH content for 25°C is kept as low as that for 15°C. Comparing Fig. 2 with Fig. 3 reveals that as the temperature increases from 15 to 25°C, the increased F content dominates the higher shift in peak position and the decrease in fwhm. As the temperature increases from 25 to 35°C, the increased bond strain that resulted from the exponentially increased deposition rate dominates the lower shift in peak position and the slight increase in fwhm.

Kawahara *et al.* also found that the fluorine content of LPD film deposited in diluted H₂SiF₆ increases when increasing the deposition temperature from 35 to 60°C.¹⁶ The increased fluorine content was attributed to the accelerating effect in polymerization of the silicic ion and the enhancement of the catalytic role of F ion. For TD-LPD film, both the F content and the OH content increase with increasing temperature. The mechanism that increases both contents is explained as follows. The intermediate species for LPD are the monomer Si(OH)_{4-m}F_m (*m* < 4) and/or the oligomer, which is formed by polymerization of monomers in the solution by the following reactions between terminating bonds²²⁻²⁴



or



Also, the Si-O networks in the films can be formed by the above polymerization reactions between the intermediates in the solution and the terminating bonds on the film surface.

According to Fig. 2, increasing the temperature exponentially increases the deposition rate. Also, with a higher temperature, the intermediates are less likely to completely react with each other or with the terminating bonds on the film surface. Finally, many terminating bonds are preserved in the film. According to the above polymerization reactions, more terminating bonds preserved in the film result in less Si-O-Si bond density. In the following, we verify this inference.

Generally, a low refractive index and a high etch rate are indicators of a low-density oxide film. In addition, the bond strain in the films can also accelerate the chemical etch rate.¹⁷ The refractive index and the P-etch rate as a function of temperature were investigated to evaluate the film. According to Fig. 4, the refractive index monotonically decreases from 1.433 to 1.423 as the temperature increases from 15 to 35°C. On the other hand, the P-etch rate monotonically increases from 16.9 to 34.8 Å/s with increasing temperature. Therefore, in terms of the refractive index and P-etch rate, the film deposited at 35°C is more porous and/or has greater bond strain than that deposited at 15°C. This finding also verifies that a greater number of terminating bonds in the film leads to a lower Si-O-Si bond density.

Figure 5 illustrates the dielectric constant as a function of deposition temperature for variously treated films. For the as-deposited film, the dielectric constant decreases from 3.56 to 3.46 as the temperature increases from 15 to 25°C. However, the dielectric constant inversely increases to 3.66 as the temperature further increases to 35°C. The dielectric constant of the film deposited at 25°C is obviously minimal. The variation in the dielectric constant of the as-deposited film is explained in terms of the F content and the OH con-

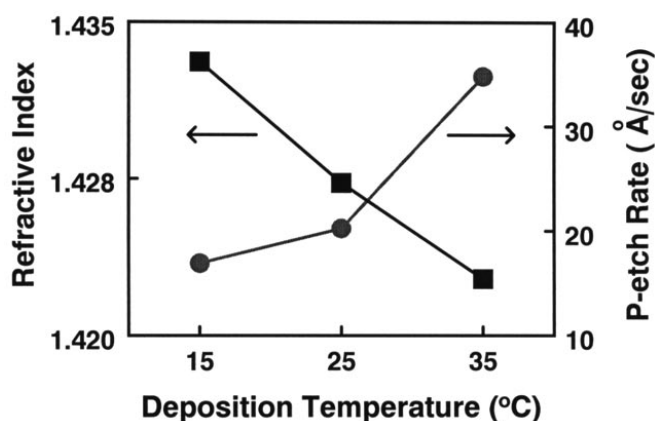


Figure 4. Refractive index and P-etch rate as a function of deposition temperature.

tent.^{2,6,19} As the temperature increases from 15 to 25°C, the increasing F content decreases the dielectric constant. While the temperature further increases to 35°C, the increasing OH content dominantly increases the dielectric constant.

Conventional FSG films easily absorb or desorb water after moisture stress or thermal annealing, leading to a variation in their dielectric constants. To evaluate the thermal stability/moisture resistance for TD-LPD FSG, this study also investigated the dielectric constant for the film after annealing at 400°C for 30 min in N₂ or after boiling in 100°C water for 1 h. According to Fig. 5, for the film deposited at 15°C, the dielectric constant increases and decreases only slightly after boiling and after annealing, respectively. For the films deposited at 25°C, the dielectric constant increases slightly from 3.46 to 3.49 after boiling, while it decreases to 3.33 after annealing. As for the films deposited at 35°C, the dielectric constant increases from 3.66 to 3.85 after boiling; while it decreases to 3.55 after annealing. Obviously, the films have a similar variation in the dielectric constant due to absorption/desorption of water. The difference in the dielectric constants between the boiled and the annealed films can reflect the total amount of absorbed water that can be removed at a 400°C anneal. In fact, the total amount of absorbed water can be divided into two parts: one is the water physically absorbed on the surface or in the pore structure, and the other is the water that is hydrogen-bonded to Si-OH bonds.^{5,12} Since the film deposited at a higher temperature is more porous and contains more Si-OH bonds, the amount of both physically absorbed water and hydrogen-bonded water is thus more significant. Obviously, the film deposited at 15°C

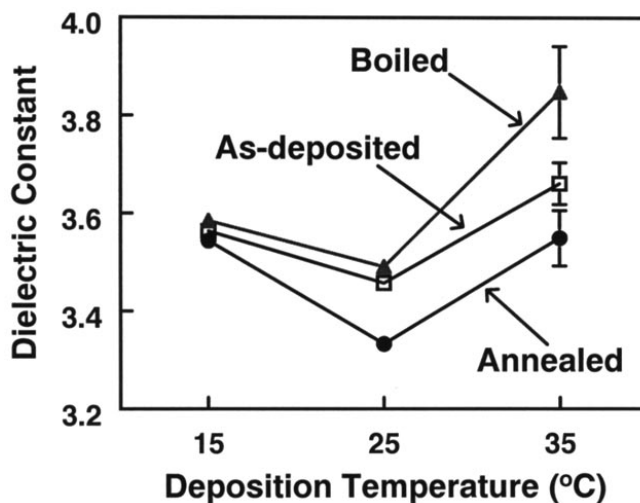


Figure 5. The dielectric constant for variously treated films as a function of deposition temperature.

exhibits the optimum stability. The condition with a deposition temperature above 35°C is unavailable owing to the relatively high dielectric constant and insufficient thermal stability/moisture resistance. The film deposited at 25°C has a rather low dielectric constant (3.46), because of a high F content (3.37%) and low OH content (0.17%). The dielectric constant can be further reduced to 3.33 with a 400°C annealing, which can remove the absorbed water only, and with almost all fluorine atoms preserved in the film. An oxide film deposited at a low temperature generally exhibits low stress as a result of the reduced thermal stress.^{13,25} Besides, the F content of oxide film also affects the intrinsic stress.¹³ Figure 6 depicts the stress as a function of temperature for the as-deposited and for the 400°C annealed TD-LPD films. For the as-deposited films the stress decreases from 63 to 43 MPa as the temperature increases from 15 to 25°C; while it inversely increases to 73 MPa as the temperature further increases to 35°C. For the film deposited around 25°C, its stress is at the minimum level. Since the incorporation of fluorine atoms can reduce the stress for the film,¹³ as the temperature increases from 15 to 25°C, the increased F content thus dominates the decrease in the stress. As the temperature further increases to 35°C, the increased bond strain resulted from the exponentially increasing deposition rate is the dominant factor in increasing the stress.^{17,26} The minimum stress for the film deposited around 25°C is thus due to high F content and low bond strain.

According to Fig. 6, after a 400°C, 30 min annealing in N₂, the stress reduces to within ±20 MPa. The net change in stress ranges from -50.3 to -63.3 MPa, *i.e.*, close to the calculated thermal stress (~-78 MPa).¹³ Besides, for the film prepared at 35°C with a relatively high OH content, the stress does not make a pronounced increase after thermal annealing, indicating that the Si-OH bond density is not high enough for neighboring Si-OH bonds to form Si-O-Si bonds. Therefore, the variation of the stress after annealing is attributed mainly to the thermal stress induced during the annealing process. Obviously, the magnitude of stress for 400°C annealed TD-LPD film is rather lower than 120~340 MPa for CVD oxide films.¹⁷

Figure 7a presents the *J-E* characteristics for the films, where negative dc voltage is applied to the Al electrode. The breakdown fields exceed 4 MV/cm, which is significantly larger than the operation field applied to intermetal dielectrics. The current densities for the films deposited at 15 and 25°C are as low as 10⁻⁹~10⁻⁸ A/cm². However, for the film deposited at 35°C, the current density is over one order of magnitude higher than those for the other samples. This

is because the film deposited at 35°C is porous and contains many defects, forming electrically active traps.

Figure 7b shows the distributions of current density at 2 MV/cm for as-deposited and annealed films. All the distributions for the as-deposited films (solid symbols) are rather narrow in current density, indicating that the as-deposited films are uniform in electrical property. After annealing at 400°C for 30 min in N₂, the median current density (*J*₅₀) for the film deposited at 15°C increases slightly from 6.9 × 10⁻⁹ to 9.0 × 10⁻⁹ A/cm²; *J*₅₀ for the film deposited at 25°C increases by a factor of five, from 4.6 × 10⁻⁹ to 2.3 × 10⁻⁸ A/cm²; for the film deposited at 35°C, *J*₅₀ increases by a factor of 5.6, changing from 3.6 × 10⁻⁷ to 2 × 10⁻⁶ A/cm². Obviously, the annealing degrades the insulating ability for the film. In particular, the degradation is rather pronounced for the films deposited at 35°C. A possible mechanism for this degradation is proposed. Since more electrically active traps in the bulk can induce a substantially higher leakage current,²⁷ the increased *J*₅₀ for the annealed film is attributed to the increased trap density. In fact, the Si-O network is not easily

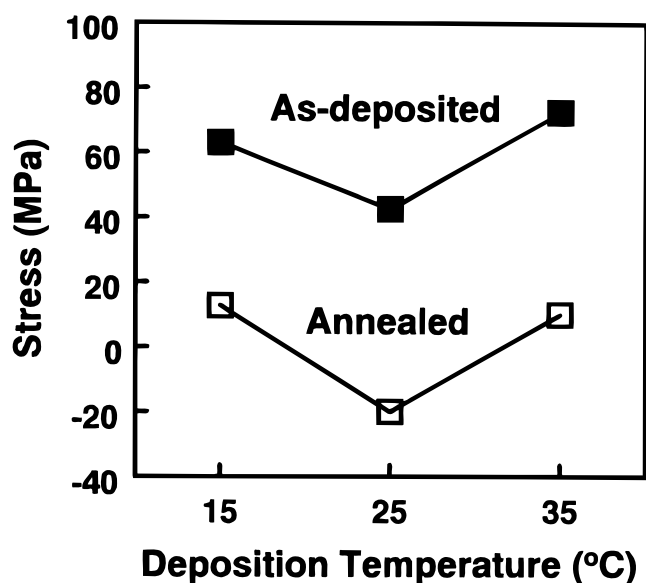


Figure 6. The stress as a function of deposition temperature for the as-deposited and the annealed films.

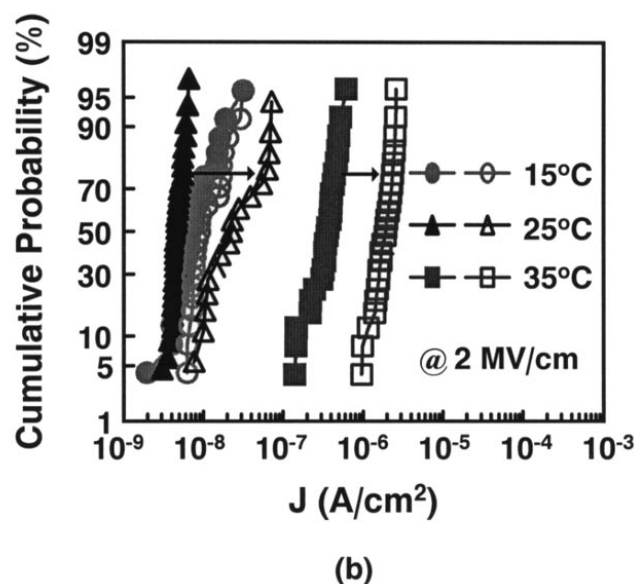
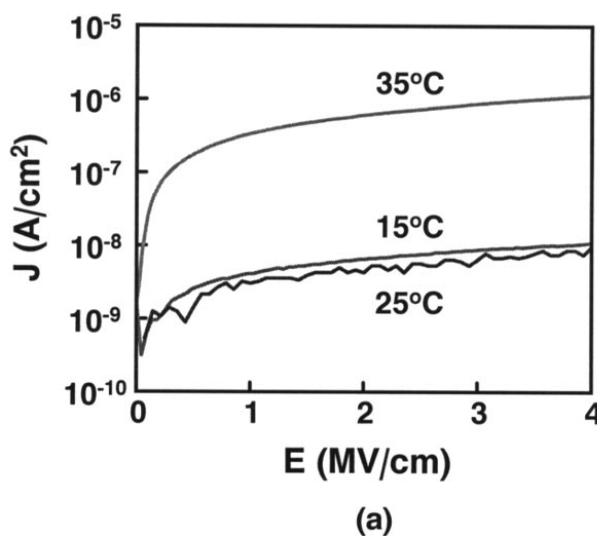


Figure 7. (a) *J-E* characteristics for films deposited at 15, 25, and 35°C. (b) Distributions of current density at 2 MV/cm for the as-deposited films (solid symbols) and the annealed films (empty symbols), with deposition temperature as a parameter.

Table I. Comparison of properties between TD-LPD FSG and PECVD FSG.

Deposition temperature (°C)	TD-LPD FSG			PECVD FSG
	15	25	35	300
F content (%)	2.56	3.37	3.49	1.44
OH content (%)	0.16	0.17	0.50	0.81
P-etch rate	16.9	20.3	34.8	27.9
Dielectric constant (1 MHz)				
As-deposited	3.56	3.46	3.66	4.00
Annealed (400°C, 30 min)	3.54	3.33	3.55	3.76
Boiled (100°C, 1 h)	3.58	3.49	3.85	5.95
Stress (MPa)	63	43	73	157
Leakage current density at 2 MV/cm (A/cm ²)	6.9×10^{-9}	4.6×10^{-9}	3.6×10^{-7}	1.5×10^{-7}

decomposed in 400°C annealing. In contrast, the weak terminating bonds can be easily removed in annealing with defects generated. Since the film deposited at 35°C has more terminating bonds, more defects must be generated after annealing, resulting in a more pronounced degradation in electrical property. According to Fig. 7a and b, the electrical insulating property and the thermal stability are optimal for the films deposited below 25°C.

To further demonstrate the feasibility of the film as an IMD, this study investigated and compared the properties of the FSG film prepared by PECVD. According to Table I, the TD-LPD film contains more fluorine atoms and less OH bonds than the PECVD film does. The P-etch rates for the both films are comparable. For as-deposited films, the dielectric constant of TD-LPD film is within 3.46~3.66, smaller than 4.0 of PECVD film. For both films after annealing at 400°C for 30 min in N₂, the dielectric constants decrease due to removal of adsorbed water. The dielectric constant varies about 0.02~0.13 for TD-LPD film and 0.24 for PECVD film. This finding confirms that the thermal stability is rather good for TD-LPD film.

After the moisture stress test in boiling water for 1 h, the dielectric constants for both films increased because of moisture absorption. The increase for TD-LPD film is within 0.02~0.19, while that for PECVD film is as large as 1.95, from the original 4 to 5.95. The excellent moisture resistance for the TD-LPD film can be attributed to a rather low OH content.

The stress of TD-LPD film is within 43~73 MPa, smaller than 157 MPa of PECVD film. The leakage current density (at 2 MV/cm) for the TD-LPD film prepared at 35°C is 3.6×10^{-7} A/cm², over fifty times of that for the films prepared below 25°C. However, it is only slightly larger than 1.5×10^{-7} A/cm² of PECVD film. Compared with PECVD film, the TD-LPD film exhibits a lower dielectric constant and a much better resistance to moisture in addition to a comparable or better insulating property.

Conclusion

This study investigated the properties of FSG films prepared by TD-LPD at various temperatures. The deposition temperature in the small range of 15~35°C significantly affects the film properties such as density, bond strain, F content, OH content, and leakage current. For as-deposited films, the film prepared at 15°C exhibits excellent stability owing to the high-density structure. The film prepared at 25°C demonstrates the minimal dielectric constant (3.46) and the minimal stress (43 MPa) due to high F content, low OH content, and low bond strain. The dielectric constant for the film prepared at 25°C can be further reduced to 3.33 by annealing at 400°C. The film deposited at 35°C is less promising as an IMD because of a relatively high dielectric constant/stress and insufficient insulating ability/thermal stability compared to the films deposited below 25°C. Compared with fluorinated IMD prepared by PECVD, the TD-LPD film exhibits a lower dielectric constant, much better resistance to mois-

ture, and comparable or better insulating property. Results of this study demonstrate that the FSG film prepared by TD-LPD is a reliable IMD with a low dielectric constant.

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