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Properties of Insulating Thin Films Deposited by RF Sputtering

Abstract: The use of RF sputtering for the deposition of thin insulating films at relatively low temperatures (50° to 500°C) has been shown to yield films of very high quality. Methods for investigating the properties of RF-sputtered films are discussed in detail, and these indicate that such films are excellent stable insulators. Potential utility as an encapsulant for transistor structures has been demonstrated. Data for RF-sputtered SiO_2 and GSC-1 (an alumina borosilicate glass) are presented as examples of the properties obtained by this technique.

Introduction

The use of RF sputtering for the deposition of thin films permits the formation of a wide variety of high quality insulating films at low deposition temperatures.[1,2,3] It is the purpose of this paper to describe the properties of RF-sputtered films and the techniques that have been used in their evaluation. We have emphasized the films obtained by the RF sputtering of fused quartz (SiO_2) and General Electric GSC-1 glass (a high melting borosilicate glass used for sealing fused quartz to pyrex). The results obtained on these films are typical of those obtained on many glasses. Comparisons between RF-sputtered glass films and films prepared by high-temperature techniques are also made.

As will become evident, a number of different techniques have been used in evaluating the films, and it should be emphasized that no single technique by itself suffices to characterize a film. These tests have included various measurements of physical parameters as well as insulation effectiveness as measured by passivation of microelectronic devices.

Techniques for film examination

- *Selective etch methods*

Difference in glass composition, bond strain, and density can be determined by means of a selective etch[4] called "P etch" (a solution of 15 parts 49% hydrofluoric acid, 10 parts 70% nitric acid, and 300 parts water), which has been effectively used in evaluating silicon dioxide and various glass films.[5,6] For example, a glass film of low density or a film with a high degree of bonding strain will etch relatively fast in P etch. The P etch rate is also very sensitive to composition.

- *Optical techniques*

Refractive indices and film thicknesses were measured by use of VAMFO, which is a specially constructed interference microscope with which it is possible to determine nondestructively the refractive indices and the thicknesses of transparent films to a precision of 0.1%.[7,8] The refractive index of a film is influenced by its density and its composition; the greater the density the greater the refractive index. The refractive index is also increased by oxygen deficiency and is affected by the presence of various contaminants in the glass. All refractive indices given in this paper will be for a wavelength of 5461Å.

- *Infra-red spectroscopy*

The simplest method for comparing RF-sputtered films with fused glass films is infrared spectroscopy. From the band positions, half-widths and intensities, information on the composition and structure of the glass can be obtained. This is best illustrated by an example. In Fig. 1, spectrum A is that of an RF-sputtered GSC-1 film while spectrum B is of a film of GSC-1 glass formed by fusing sedimented powder.[9] The band at 1390 cm^{-1} is attributed to B-O stretching and the band at 1070 cm^{-1} to Si-O stretching. The relative intensity of these two bands is almost the same, indicating that the relative amounts of boron oxide to silicon dioxide are not much different in

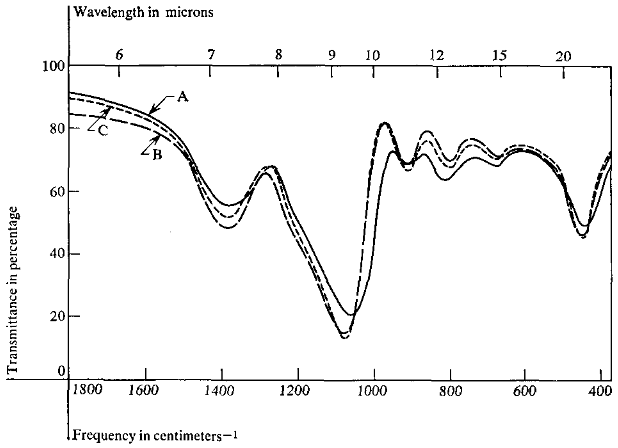

Figure 1 Infrared spectra for (A) RF-sputtered GSC-1 on ~ 100°C silicon substrate (0.809 μ); (B) GSC-1 film formed by fusing sedimented powder (0.778 μ); (C) GSC-1 film from A after heating in dry N_2 for 5 min. at 965°C (0.786 μ).

Table 1 Comparison of RF-sputtered SiO_2 films with a thermally grown SiO_2 film (~ 5700 Å thick).

Sample	*Substrate temp.*	*Optical Density of OH at 3600 cm⁻¹*	*Si-O stretching absorption bands*		
			Position $\nu_a \sim 1100$ cm^{-1}	*Half-width ν_a*	*Position $\nu_b \sim 800$ cm^{-1}*
RF SiO_2	100°C	0.0026	1057	107	816
RF SiO_2	450°C	0.0004	1072	98	813
Thermal (DWD)	980°C	——	1087	81	805

Table 2 Refractive index and P etch rates of sputtered SiO_2 films.

Sample	*Substrate temp.*	*Deposition rate Å/min.*	*R.I. at 5461 Å*	*P etch rates (Å/sec.) at 25°C*
RF SiO_2	100°C	1186	1.476	10.2
RF SiO_2	450°C	865	1.473	4.6
Thermal (DWD)	980°C	—	1.461_8*	2.0

* *Reference* 8.

these two films. On the other hand, in the spectrum of the sputtered film the Si-O band position is at longer wavelength and the band half-width is broader. On the basis of previous studies on SiO_2 films,[5] it is concluded that this is due to bond strain and lower density in the sputtered film as compared to the fused GSC-1 film. Furthermore, the band at 915 cm^{-1} is much weaker in the RF-sputtered film than it is in the fused GSC-1 film. It has been found that this band is much more sensitive to structure than most of the other bands in the spectrum. This band is due to a stretching vibration of the Si-O-B group and this sensitivity of intensity to structure has been observed previously in comparing the spectra of powdered glasses with those of fused glasses.[6] Spectrum C was obtained after heating the sputtered film for five minutes in dry N_2 at 965° C, which is above the annealing point but below the softening point of GSC-1 glass. This heat treatment densified the film 2.8% (thickness decreased to 0.786μ from 0.809μ). Furthermore, before heating the sputtered film had a refractive index of 1.478, whereas after heating it was 1.466, which is practically the same as the refractive index of 1.467 measured on a fused GSC-1 film. The larger initial refractive index can be attributed to a slight oxygen deficiency. On the sputtered film the P etch rate was 17.7 Å/sec prior to heating and 8.3 Å/sec after heating, which is practically the same as the P etch rate of a fused film. The most striking change in the infrared spectrum is the increased intensity of the 915 cm^{-1} band. In addition, the Si-O absorption band has shifted and has changed so as to be more similar to that of a fused film. The close similarity in the spectra between the heated RF-sputtered film and the fused GSC-1 glass film shows that the composition of both films is approximately the same, whereas the differences in the initial spectra indicate that the structure of the as-deposited RF-sputtered glass was not exactly the same as that of the fused glass.

The position of the Si-O stretching band near 1100 cm^{-1} shifts to higher frequency as the degree of polymerization of the SiO_4 tetrahedra in the silicate lattice is increased.[10,11] This shift to higher frequency has also been explained, somewhat similarly, in terms of densification of the silicate structure.[5] In addition, it has been observed that bond strain will shift the Si-O band towards lower frequency.[5,12] The explanations are reconcilable in that porosity and strain can limit the degree of polymerization of the SiO_4 tetrahedra. For the general case of RF-sputtered films no single explanation is sufficient. In many cases, a decrease in film thickness on heating indicates the occurrence of densification. However, the degree of densification is, in general, significantly smaller than that observed with SiO_2 films deposited by other low temperature techniques, such as pyrolysis of Si $(OC_2H_5)_4$ or electron-gun evaporation of SiO_2. Furthermore, with many RF-sputtered films, significant spectral changes take place on heating without any pronounced thickness change, showing that some other type of structural modification has occurred. For the particular case shown by Fig. 1, densification has accompanied the structural modification indicated by the spectral changes.

Effect of substrate temperature during deposition

Substrate temperature was found to affect film properties more than any other deposition parameter. As expected,

Table 3 Effect of mild heating (15 min. dry nitrogen at 520°C on RF sputtered SiO_2 films).

	Refractive index (5460 Å)		*Infrared examination (film thickness* 5600–8500 Å)			
			1100 *cm^{-1} band position*		*Half-width*	
Type sample	*Initial*	*Average change due to heating*	*Initial (cm^{-1})*	*Shift due to heat*	*Initial (cm^{-1})*	*Decrease due to heat*
RF Sputtered:						
Cold (50–100°C)	1.473 to 1.479	-0.011 ($d > 2\mu$) -0.022 ($d < 1\mu$)	1057 to 1064	+5 to 7	105 to 107	2 to 6
Hot (400–530°C)	1.467 to 1.475	-0.002	1070 to 1074	+1 to 3	98 to 103	1
Thermal SiO_2	1.461_8*	—	1087 to 1091	—	81 to 86	—

* *Reference* 8.

faster deposition rates tend to give films of lower density; however, the effect of deposition rate was found to be relatively small for the higher rates which are normally used for routine deposition.[2]

RF-sputtered SiO_2 films deposited at a substrate temperature of ~450°C resemble thermally grown SiO_2 films more closely than do films deposited at ~100°C. This is summarized in Table 1 where two RF-sputtered SiO_2 films approximately 5700Å thick are compared to a thermally grown SiO_2 film of the same thickness. The substrate temperature was measured with a fine-wire thermocouple which was brazed onto the front of a control wafer. It is estimated that the measured temperature might be as much as 50°C lower than the actual temperature.

The designation DWD for thermally grown films means that the films were grown by exposure of a silicon wafer for about fifteen minutes to dry oxygen, followed by steam for a period sufficient to grow the proper thickness film and finally to dry oxygen for at least one half hour. The films were grown at 980°C. As can be seen in Table 1, the amount of adsorbed water, as indicated by the intensity of the hydrogen bonded silanol band at 3600 cm^{-1}, is significantly less in films deposited at substrate temperatures of 450°C than in films deposited near or below 100°C.

Furthermore, the difference between the Si-O absorption band positions for the sputtered film and for the thermally grown oxide is greater for the low-temperature deposited film than for the high-temperature film. The spectra also show greater differences in the band half-widths between the low-temperature film and thermally grown oxide than between the high-temperature film and thermally grown oxide. It should be noted that, in making these comparisons, films of comparable thickness should always be used, since both the band position and the half-width of the spectrum vary slightly with the thickness of the film.[5] Even though the lower-temperature RF-sputtered films do not resemble the thermally grown films as closely as do the high-temperature films, they are still much better in quality than SiO_2 films obtained by other low-temperature deposition techniques, such as evaporation or pyrolytic decomposition.

Evaluations were also made on RF-sputtered SiO_2 films of greater thickness. In this case, it is more difficult to compare the infrared spectra because the high absorption band intensity reduces the accuracy of band position and half-width determinations; therefore comparisons on the thicker films were made by measuring the refractive indices and P etch rates of the films. These results are summarized in Table 2. Film thicknesses were about 2.4μ. Again, a comparison shows that the 450°C substrate film is closer to thermally grown SiO_2 than the 100°C substrate film. The P etch rate of the 100°C film is seen to be much higher, indicating that the film is more strained and/or less dense. The higher refractive index of the films compared to that of the thermally grown silicon dioxide films is believed to be due mainly to a slight oxygen deficiency, which we shall discuss in the next section.

Many cold and hot substrate samples of RF-sputtered SiO_2 films have been investigated. Part of this investigation has included an examination of the effect of a relatively mild post-heat treatment. The initial heat treatment varied from 10 to 20 minutes' exposure to dry nitrogen at 500 to 540°C. We were especially interested in the possibility of structural modifications at these temperatures. This treatment was not sufficient for significant densification or drying (removal of hydroxyl groups), but the treatment was sufficient to show differences between the hot- and cold-sputtered SiO_2 films. These results are summarized in Table 3.

As deposited, cold-sputtered films had higher refractive indices than hot-sputtered films. Heat treatment reduced the refractive indices of the cold-sputtered films, especially the thinner ones. The refractive indices of the hot-sputtered films were reduced only slightly. These differences could be attributed to the entrapment (or adsorption) of gases or volatile impurities in the less dense cold-sputtered films. Because of diffusion effects, these substances are removed more readily from the thinner films on heating. During the heat treatment in nitrogen some of the film is probably oxidized by the slight amount of moisture present in the film. This oxidation would decrease the refractive index. Since the cold-sputtered films have more moisture, as indicated by the intensity of the 3600 cm^{-1} band, one might conclude that the greater decrease of the refractive index of the cold-sputtered film on heating in nitrogen is due to this effect. But this would not explain the greater decrease of the refractive index of the thin films of the cold-sputtered SiO_2 as compared to the thick films, which can be explained by a volatile impurity hypothesis. Sufficient heat treatment would also tend to densify the films; this would be reflected by a refractive index increase. It is concluded that all these effects occur to some extent.

The infrared spectra show that the low-temperature films are more amenable to structural modification by this relatively low-temperature treatment. The heat treatment is sufficient to shift the 1100 cm^{-1} band 5 to 7 cm^{-1} towards higher frequency with a corresponding half-width decrease of 2 to 6 cm^{-1} whereas, with the high substrate temperature samples, the spectra are closer to that of thermally grown SiO_2 and the subsequent changes on additional heating are not as pronounced. Although the post-deposition heat treatments of the "cold" samples were in some cases for longer times and at higher temperatures than the deposition times and temperatures of the films sputtered on hot substrates, the infrared spectra show that the latter, even without any post-deposition heat treatment, are closer to thermal oxide than the cold-substrate films with post-heat treatment. This implies that the equilibrium conditions can be reached more readily if the film is heated as it grows.

Thickness changes on heating show that the cold-sputtered films are slightly less dense than the hot-sputtered films. This is also substantiated by the greater intensity of the 3600 cm^{-1} band due to hydrogen bonded silanol groups in the untreated cold-sputtered films.

RF-sputtered SiO_2 films could be made virtually indistinguishable from thermally grown SiO_2 by high-temperature baking in an oxidizing atmosphere. The 5700 Å sputtered films associated with Table 1 were exposed to oxygen for eight minutes at 990°C. The infrared spectra of both the cold- and hot-substrate films were practically indistinguishable from that of thermally grown SiO_2. The half-widths of the main absorption band were 81 cm^{-1}, the same as for thermally grown films; the absorption bands were at 1086 cm^{-1}, only 1 cm^{-1} removed from that of thermally grown oxide; the measured refractive indices were 1.459 and 1.462 as compared to 1.461_8 for thermally grown oxide[8]; and the P etch rates were practically the same as for thermally grown SiO_2.

Oxygen deficiency

Usually no significant oxygen deficiency is observed in RF-sputtered SiO_2 films. Where it has been detected, it is indicated by:

(1) the shift of the 1100 cm^{-1} band to lower frequencies (longer wavelengths),
(2) the presence of a band near 875 cm^{-1} due to Si_2O_3,
(3) slow P etch rate,
(4) high refractive index, and
(5) growth of the film during a subsequent oxidation treatment.

The Si-O stretching band in the spectrum of silicon monoxide[5,13] is near 1000 cm^{-1}, in Si_2O_3[5,14,15] near 1040 cm^{-1} and in thermally grown silicon dioxide[5] near 1085 cm^{-1}, depending on the film thickness. The use of the band position for determining oxygen deficiency is not satisfactory in itself, because of the effects of density and bond strain, as previously discussed.

In addition to the shift of the main Si-O stretching absorption band with the state of oxidation, a band at 875 cm^{-1} is observed in the spectra of Si_2O_3 films.[5,14,15] In the case of electron gun-evaporated SiO_2 films, a weak band at this position was observed after exposure of the films to high humidity or to slow heating up to 600°C,[12] but the band intensity was relatively weak, indicating only a slight oxygen deficiency. This band has not been observed in the spectra of the RF-sputtered SiO_2 films and therefore it is concluded that the oxygen deficiency of the sputtered films is even less.

Determination of oxygen content in sputtered SiO_2 films

If density corrections can be made, then the degree of oxygen deficiency can be determined by the thickness increase of the film on oxidation. Let d_{SiO_x} represent the film thickness of the RF-sputtered film and d_{SiO_2} represent the thickness of the same film after oxidation. The ratio of film thicknesses is given by:

$$r = d_{\mathrm{SiO}_2}/d_{\mathrm{SiO}_x} = (M_{\mathrm{SiO}_2}\rho_{\mathrm{SiO}_x})/(M_{\mathrm{SiO}_x}\rho_{\mathrm{SiO}_2}),$$

where ρ_{SiO_x} is the density of the RF-sputtered films, ρ_{SiO_2} is the density of the SiO_2 film, M_{SiO_2} is the molecular weight of SiO_2, and M_{SiO_x} is the molecular weight of SiO_x. The density of fused quartz is 2.20 g/cc and the density of silicon monoxide is 2.18 g/cc. Since the densities are nearly the same, it will be assumed that the density of SiO_x, the sputtered film, is practically the same as SiO_2 and the above equation reduces to

$$r = M_{\mathrm{SiO}_2}/M_{\mathrm{SiO}_x} = 60.1/(28.1 + 16.0x),$$

$$x = (60.1 - 28.1r)/16.0r.$$

Use of this formula is most accurate on hot-substrate RF-sputtered films since the correction for density changes in such films is small. Such corrections can be made because there is a relationship between the 3600 cm^{-1} band intensity (due to hydrogen bonded hydroxyl groups) and the film density.[5] The corrections given in Table 4

Table 4 Oxygen deficiency as determined by film thickness ratios of oxidized to non-oxidized RF-sputtered SiO_2 films.

Substrate temp., °C	*Initial thickness*, (Å)	*Optical density at* 3600 *cm*$^{-1}$ (SiOH)	*Density correction*, (Å)	d_{SiO_2} *Corrected initial thickness*, (Å)	d_a *Oxidized thickness*, (Å)	d_o *oxide on bare Si*, (Å)	d_{SiO_2} *corrected oxidized thickness*, (Å)	*r*	*x*
~50	8350	~0.0053	290	8015	8147	615	8124	1.014	1.95
~100	5662	0.0026	143	5519	5550	360	5538	1.003	1.99
~450	5675	0.0004	22	5653	5750	210	5746	1.016	1.94
~500	9263	0.0011	60	9203	9375	650	9352	1.016	1.94

are based on comparisons between high quality non-densified and densified pyrolytic films. The films were oxidized either by exposure to dry oxygen at 1200°C for approximately five minutes or to wet oxygen followed by dry oxygen for a total of 8 minutes at 990°C. Since these films were deposited on bare silicon, allowance was made for the increase in film thickness due to oxidation of the silicon wafer. For all practical purposes the corrected film thickness is given by $d = \sqrt{d_a^2 - d_0^2}$ where d_a is the final measured film thickness and d_0 is the oxide film thickness which would be formed on a bare silicon surface due to the oxidizing treatment. After oxidation, the sputtered films were indistinguishable from thermally grown SiO_2 as determined by infrared measurements, refractive index, and P etch rates. The results, as given in Table 4, show that most of the RF-sputtered SiO_2 films as formed initially are only slightly oxygen deficient, having a formula of $SiO_{1.95}$ (i.e., $x = 1.95$).

Close examination of Table 4 shows why we claim greater accuracy with the hot-substrate RF-sputtered films. Although the density corrections based on the optical densities of the 3600 cm^{-1} hydrogen-bonded SiOH absorption band are only approximately accurate, the correction itself is so small that the error introduced by the approximation is even smaller. With the hot-sputtered films these corrections are significantly smaller than with the cold-sputtered films and the error introduced by the approximation is insignificant. It is obvious that density corrections must be applied to the cold-sputtered films, since the final film thicknesses d_a are less than the initial thicknesses. In these cases, if these corrections were not made, we would obtain values for x greater than two. If we had neglected these density corrections on the 450° and 500°C substrate films we would have obtained 1.95 and 1.96 for x.

Mainly because the sputtered film is not exactly the same as thermal SiO_2 initially and only becomes so with oxidation, the very small thickness correction, $d_a - \sqrt{d_a^2 - d_0^2}$ for the oxidation of the silicon underneath the film is also only approximate. However, the correction is again so small that the error introduced by the approximation is insignificant. If we did not correct for the oxidation of the silicon underneath the film, which is equivalent to making the extreme assumption that the oxygen does not penetrate the film, we would have obtained 1.94, 1.98, 1.94, and 1.93 for x. These values are virtually indistinguishable from the corrected results. On the other hand, the oxidation of the underlying silicon can not be significantly greater than that given by our correction for then $r < 1$ and $x > 2$.

Boron deficiency

In fabricating targets for RF sputtering of glass films, special care must be taken with mixed-composition glasses containing volatile components which may fractionate. The reason for this can be seen quite clearly with some RF-sputtered GSC-1 glass films. Spectrum A of Fig. 2 shows an RF-sputtered film made from a GSC-1 glass plate which, in effect, had been fire polished during fabrication. Spectrum B is of a film sputtered from a

Figure 2 Infrared spectra showing effects of target surface removal and run-in time on mixed-composition glass films: (A) GSC-1 film RF-sputtered from a fire-polished GSC-1 plate; (B) film sputtered from same plate after removal of the outer layer of the plate by grinding; (C) film sputtered from same plate after grinding and run-in period.

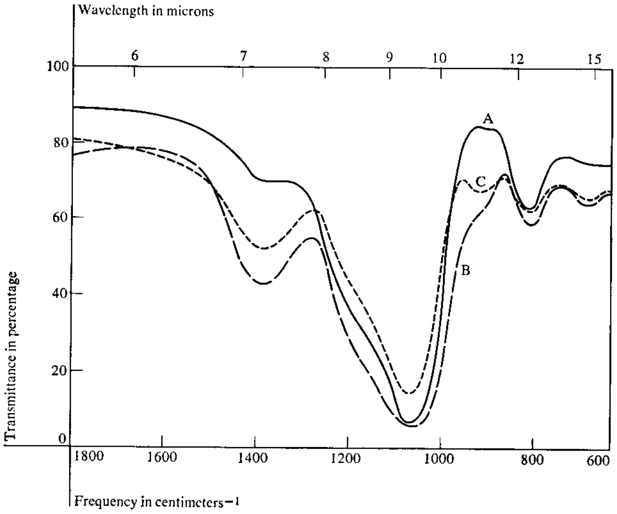

Table 5 Transistor reliability with sputtered GSC-1 films.

Glass	*Time, hours*	*Sample size*	*Test condition*	*Number of life failures*	
				Reverse	*Forward*
GSC-1 hot	2000	17	200°C back bias	1	0
GSC-1 cold	2000	10	200°C back bias	0	0

target of GSC-1 whose outer layer had been ground away. It is quite obvious from a comparison of the intensity of the band at 1390 cm^{-1} that the film corresponding to spectrum A is boron deficient, so presumably the outer layer of the GSC-1 target was boron deficient. The target as a whole was not boron deficient, as shown by the fact that the spectrum of a sedimented and fused film formed from a ground portion of the target was similar to the spectra of other fused GSC-1 glass films. The boron deficiency in the fire polished plate arises from the fact that, near the surface of fired glass objects, the boron composition is low. This has been demonstrated on fused glass films which were fired for an extended period at high temperature and then analyzed by P etch rate techniques.[6] In such cases the etch rate near the surface is significantly less than the etch rate deeper in the film. Therefore, in fabricating a target of a high-temperature borosilicate glass such as GSC-1, the boron will out-diffuse from the glass since the boron oxide is much more volatile than the other components of the glass. Thus in many of these targets it is necessary to remove the outer layer of material to obtain the desired composition in the RF-sputtered films. It is also good practice to have some "run-in" time for RF sputtering whenever a target is used for the first time, as shown by comparing spectrum B with spectrum C. The boron composition of the film with greater "run-in" time (spectrum C) is closer to that of GSC-1 glass.

Non-uniformity across the target can also cause variation in the properties of films formed on different substrates but during the same run. This occurred in a target that developed a hot spot during sputtering. Infrared spectroscopic examination at three regions across the wafer showed that the region of the sputtered film closet to the hot spot had less boron (weaker 1390 cm^{-1} band) than the center of the wafer, while the region furthest from the hot spot contained the most boron.

Application to semiconductor devices

An important application of an insulating film is as an encapsulant for semiconductor devices. Accordingly, various sputtered glasses were applied to standard planar transistor wafers using a variety of different sputtering parameters. Device wafers containing aluminum metallurgy were coated with sputtered films and then subjected to further standard processing and evaluation steps. All wafers processed were split in two in order that half of the wafer could be protected with a layer of sedimented glass[9,16,17] to provide control devices for the sputtered units.

An initial objective of the device evaluation effort was to rapidly determine whether any gross deterioration of electrical parameters would occur due to the application of the sputtered glass film to the device surfaces. GSC-1 was chosen as a test vehicle for evaluating this application both because of its excellent physical and chemical properties and because it had previously been used in fritted form in other experiments and consequently was somewhat familiar to us. The sputtered glass was deposited on two wafers at a temperature of approximately 450°C and on a third wafer whose temperature was kept below 150°C during deposition. Film thicknesses were about 2.5μ. After deposition, the glass films were etched, the transistors were electrically probed (and device parameters compared to the pre-glassing values), diced, mounted on TO-18 headers and wire-bonded before being placed on life test at 200°C back bias (3V emitter, 10V collector). Twenty-seven transistors from these wafers were placed on test and after 2000 hours the only failure observed was on one transistor which showed steadily increasing emitter leakage current. Characteristics measured included BV_{CBO}, BV_{EBO}, I_{CBO}, I_{EBO}, I_{CEX}, V_{FE}, V_{FC}, FV_{CBO}, FV_{EBO}, V_{CE} and β (these parameters were measured on all of the devices placed on life test during this study). Reliability data on these units are summarized in Table 5.

Based on these encouraging results it was decided to evaluate the use of sputtered SiO_2 for device passivation. SiO_2 has the advantages of being very easily available and since it is a single-component glass it is easier to sputter reproducibly. For these reasons SiO_2-coated wafers were prepared and placed on life test, using both hot (~450°C) and cold-sputtered SiO_2. Reliability data on these units are summarized in Table 6. These data indicate that sputtered SiO_2 can be applied to device wafers with the resultant devices having an electrical reliability equivalent to that achieved with the sedimented glass devices.

Mechanical properties and stress in RF-sputtered films

Generally, it has been found that RF-sputtered films have excellent mechanical properties. The films are tough and adhere well to the surface upon which they have been deposited. Scribing the film gives sharply defined scratch marks without the formation of hairline cracks. Ultrasonic cutting of wafers coated with sputtered films has given good sharp edges, indicating good adhesion of the glass.

Table 6 Transistor reliability with sputtered SiO_2 films compared with a borosilicate glass.

Film type	*Hours on test*	*Sample size*	*Type of test*	*Incidence of failures*	
				Reverse	*Forward*
SiO_2 hot	1000	76	150°C back bias	0	0
		76	T & H back bias	2	0
SiO_2 cold	1000	72	150°C back bias	0	0
		72	T & H back bias	0	0
Sedimented borosilicate glass	1000	116	150°C back bias	6	1
		116	T & H back bias	0	0

Another test, which gives a more quantitative measure of mechanical integrity, is the micro-hardness or indentation test. A specially shaped point is pressed against the film, and, successively, weights are added to it until the film fractures. Table 7 shows some of the results obtained for various fired and sputtered films. It was observed that when the sputtered glasses break, a very clean break takes place. This is also an indication of the good adhesion between the glass and the oxide film on the wafer.

The good mechanical properties of these sputtered glass films can be explained by the fact that the films are under compressive stress. This is because the glasses used have a coefficient of thermal expansion smaller than silicon.

The good adhesion of the sputtered glass is caused by the relatively high energies of the sputtered species and by the continuous cleaning of the substrate surface during sputtering by the glow discharge itself. This particular feature is typical of sputtered films in general.

Stress measurements[18] showed compressive stresses of 1.6×10^9 and 0.5×10^9 dynes/cm^2 for 2μ thick SiO_2 films deposited at 450°C and 100°C respectively. A 10-minute heat treatment at 500°C gave a further increase in the stress of the cold-deposited film to 0.8×10^9 dynes/cm^2, while the stress in the film deposited at 450°C did not change. This amount of compressive stress is accounted for almost completely by the thermal mismatch indicating essentially zero intrinsic stress in the films.

Conclusions

The data obtained are indicative of the high quality films that can be obtained using RF sputtering. Evaluation of RF-sputtered silica and borosilicate glass films by the physical and chemical techniques described in this paper show the potential utility of these films as insulators and coatings for electrical devices. It should be pointed out that the materials discussed here are only two of a wide variety of films that have been deposited by RF sputtering.

Table 7 Microhardness data.

Glass	*Weight at which fracture takes place (grams)* *Over oxide*	*Over aluminum lands*
Fired GSC-1	70–80	—
Sputtered GSC-1	140–160	60
Sputtered Quartz	140–160	60

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