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Conformal deposition of LPCVD TEOS.

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ABSTRACT

The step coverage of dielectrics is important for the microelectronics industry and critical to Micro-machined products and High Voltage MEMS drivers. The techniques used to fabricate MEMS structures require void free refill processes and even film deposition along deep trenches to protect against etch chemistries. High voltage drivers used to actuate MEMS devices benefit from dielectric isolation, which reduces the need for large tub formation between devices. It also enables "system on chip" solutions for MEMs devices and protection against voltage spikes. This paper presents a process developed at Analog Devices Belfast that enables an LPCVD TEOS furnace to perform a highly conformal trench refill without equipment modification. The conformality is over 95% for 20um deep trenches and maintains a conformality greater than 85% in 50um deep trenches. This compares with 75% conformality which is considered excellent for 20um trench refills obtained using previous[1,2] LPCVD TEOS processing. The process is shown to have benefits in conformality, breakdown voltage, and stress over standard trench fill processes including Ozone TEOS. The densification of the TEOS film has been optimized for electrical parameters using CV and IV techniques, while XPS, FTIR and spectroscopic ellipsometry are used for physical characterization. Stress is a very important parameter for micro-machining and the conformal TEOS has a film stress which is tensile 30-40MPa as deposited and compressive 100MPa after densification. The breakdown voltage has been measured at 8.5MV/cm compared to 7.5-9MV/cm for a typical densified TEOS film and the refractive index is 1.456 compared to 1.465 for a thermal oxide. Analog Devices Belfast is part of the Micro-machined Products division and provides SOI and customized SOI for the MEMs and IC market.

Keywords: Conformal Oxide, trench refill, dielectric isolation, High voltage, MEMS drivers.

1. INTRODUCTION

MEMs devices require a dielectric to isolate deep trenches with up to a 15:1 aspect ratio in some applications. Most literature promotes doped TEOS and Ozone TEOS to overcome the difficulties of step coverage. LPCVD TEOS has been used in industry for more than thirty years and the equipment and processes are well established. It has many benefits over other methods of producing SiO_2 films, for example production cost, low stress and conformal properties. Undoped TEOS does not have the application restrictions of doped BSPG-TEOS (Borophosphosilicate Glass) and does not require additional expensive equipment as with Ozone TEOS.

The conventional LPCVD TEOS process at 700oC and 400mTorr produces a film, which is only around 50% conformal and causes bread-loafing which results in voiding at the mouth of deep trenches. TEOS is reaction rate limited under these conditions and the difference between partial pressures on the wafer surface and in the trench causes the variation in conformality. We initially developed the process to give 75% conformality, which is sufficient for application to high voltage SOI products (4), but the introduction of MEM applications with deep trenches required a further improvement in conformality. MEMs devices require an oxide filled trench, which under standard LPCVD TEOS conditions, suffers from voids at the mouth of the trench. This voiding results in problems with subsequent etch and release processes allowing preferential etching along the oxide interface. Trench voiding also affects topology and is problematic for subsequent layer formation.

The aim of this work was to develop the LPCVD TEOS process for deep trench refilling and to characterize the annealing process, used for post deposition densification of the TEOS films. This has incorporated evaluation of the Ozone/TEOS process (3) and comparison of this with the existing and optimized LPCVD TEOS processes.

2. PROCESS OPTIMIZATION

In this paper, a Design of Experiments (DOE) is used to design an LPCVD process to produce a void free deep trench refill. A horizontal 6" LPCVD furnace was used with TEOS injected by direct draw and controlled using a Schumacher Vmdot. Pressure control was achieved with a MKS butterfly valve and a diaphragm pressure gauge on the source flange. A one hundred and twenty-five wafer load was used on non-contiguous quartz boats. A Mass-vac three stage multi-trap was used for abatement. Ozone TEOS samples were produced using a WJ-1000 NSG TEOS reactor.

Since it is known that increasing the process pressure increases the conformality of the deposited TEOS oxide(1), the pressure was increased to near the documented limit of TEOS processes(2). By reducing the deposition rate of the TEOS oxide and increasing the residence time in the reactor, the difference in TEOS partial pressures in the trench and at the wafer surface can be reduced. This gave an increase in conformality from the standard processes but uniformity was degraded. A DOE was used to factor the best process for TEOS conformality using pressure, temperature and deposition rate. Different reactor conditions were used to arrive at the optimized process using a full factorial design. The process was characterized for run to run, within wafer and across the furnace load, for conformality and thickness. The conformality was measured on 3-4um wide, 20um deep trenches and 50um deep trenches, which were dry etched at Analog Devices using an STS ICP tool and imaged using a JEOL SEM. The post deposition anneal temperature is a critical parameter in TEOS layer formation, which are used to densify the films, remove volatiles and improve the films properties. Both rapid thermal annealing (RTA) and conventional furnace annealing were investigated and extensive physical and electrical characterization was performed to optimize annealing conditions.

The electrical parameters were characterized by C-V and I-V techniques using doped Polysilicon contacts to make 0.785mm^2 MOS capacitors. The capacitors were used to test oxide charge, breakdown voltage and leakage current. The physical characteristics of the optimized process were compared with the DOE designed processes, as well as the existing "standard" 75% conformal process and thermal oxide using SE, FTIR and XPS analysis. Spectroscopic ellipsometry (SE) spectra were measured over the UV-visible range (250-850nm) using a Sopra GES5 SE instrument. Film thicknesses and % void contents were obtained by best fits to the data with a SiO_2 reference spectrum (Palik). Fit quality was judged to be very good in all cases. Refractive index (n) spectra were derived from Cauchy fits to the data, and were supported by point-by-point calculations in some cases. X-ray photoelectron spectroscopy (XPS) was carried out using a VG Escalab II instrument with MgKα incident radiation (1253.6keV) and a photoelectron take-off angle of 75° to the sample surface. Wide scan spectra were taken, followed by detailed scans of the Si2p, O1s and C1s photoelectron peaks. XPS signals derive from the surface 5-10nm of the layers. Fourier transform infrared spectroscopy (FTIR) spectra covering the range 400-4000cm^{-1} were obtained in transmission mode using a Perkin-Elmer FTIR spectrometer. After conversion to absorbance units, the background spectrum from an Fz as-received wafer, , was subtracted. The TEOS layers were also deposited on Fz material.

3. RESULTS

3.1 Conformality Results

Repeated characterization results showed a within wafer uniformity of better than ±3% and a run to run uniformity of ±5% for a 125 wafer, 150mm diameter horizontal furnace using the optimized process. This compares to a within-wafer uniformity of ±2% and a run to run variation of ±3% for a conventional TEOS process, (400mTorr, 700oC). The Ozone TEOS samples exhibited a within-wafer uniformity of <±4%. The high conformality TEOS process has not yet been fully optimized for run to run repeatability, and further statistical control should achieve ±3%. SEM measurements on 20um deep trenches indicate that a conformality of 95% -100% can be achieved, as shown in Figure 1. An important effect of the process is that bread-loafing is eliminated, so that trench profiles are not as critical in achieving void free trench refill. This avoids the need for two step or tulip etches. The conformality has also been measured within-wafer and across the furnace load as well as run to run, indicating a variation of 10% in conformality with a 125 wafer load, for a 20um deep trench. Further characterization with 50um deep trenches indicates a drop off to a minimum 75% conformality at the source end of the furnace, but greater than 89% conformality along the rest of the furnace. The drop off in conformality may be due variations in the SEM measurements, or to pressure variation across the furnace, since the pressure increases towards the source or pump end. Deeper trenches will reduce the conformality further so that deposition thickness will have to be increased to counteract this.

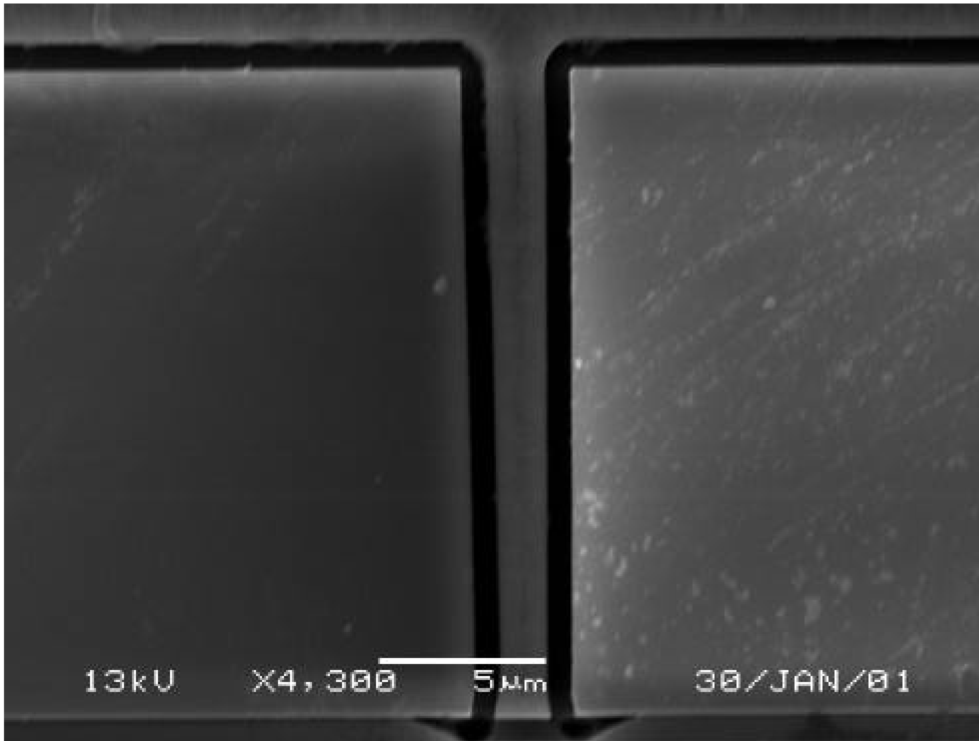


Figure 1 95% conformal 1um oxide LPCVD TEOS layer on a 20um deep, 3um wide trench with Polysilicon refill on SOI.

With this process oxide filled trenches are repeatable without voiding at the mouth of the trench, as shown in Figure 2. Trenches up to 4um wide can be filled with TEOS only, and although thicker films are possible, cracking will likely occur.

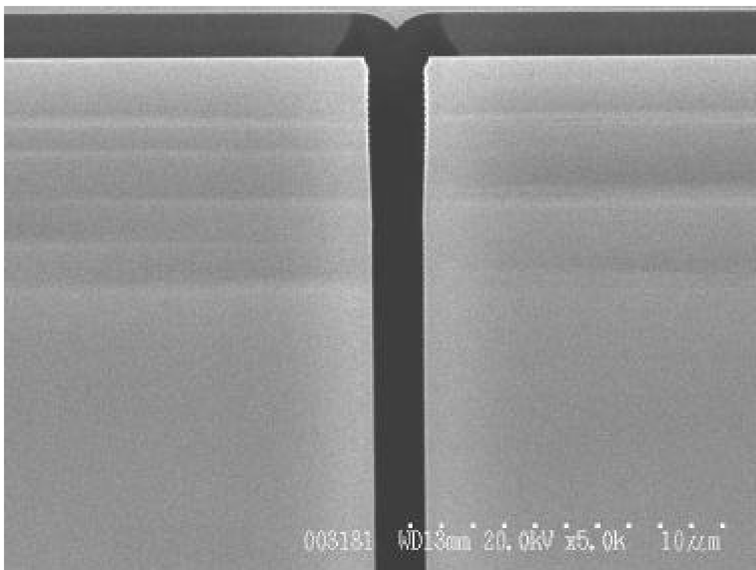


Figure 2 Oxide filled Trench with LPCVD TEOS.

3.2 Spectroscopic ellipsometry Results

Table 1 lists the layer thicknesses, void fractions (i.e. SiO_2 density lowering) and refractive index values at 634nm derived from the SE data from three iterative TEOS processes of different conformalities. Values of thermal oxide, grown at 850oC, are also included for comparison with densified TEOS films to investigate the effects of annealing. This table shows that the optimized process has the highest refractive index after annealing from the DOE iterations, which correlates with a higher film density. This should result in a more reliable breakdown voltage. The data also indicates that 1050oC is the optimal temperature for densification and RTA annealing is less effective than conventional processing. The TEOS from DOE2 gave the highest void fraction and %voids, but has lower conformality. Figure 3 is plotted to show the direct correlation between void fraction and reduction in the value of n. There are systematic tendencies for the thicknesses of TEOS layers to decrease with increasing densification or rapid thermal anneal temperature. Considerably higher RTA temperatures are required to achieve the same thickness reduction as achieved in the standard densification anneals. The TEOS films from DOE2 are an exception in showing an apparent increase in thickness on annealing. The TEOS thickness given in Table 1 indicates matter thickness and there are signs that the most marked reduction in this occurs during the lowest temperature anneal. This correlates with the disappearance of hydroxyl groups in the IR spectra, (c.f. section 3.4), and so is consistent with dehydration as the main loss of material occurring at this stage. The difference in matter thickness between TEOS film types is due to targeting of the individual processes.

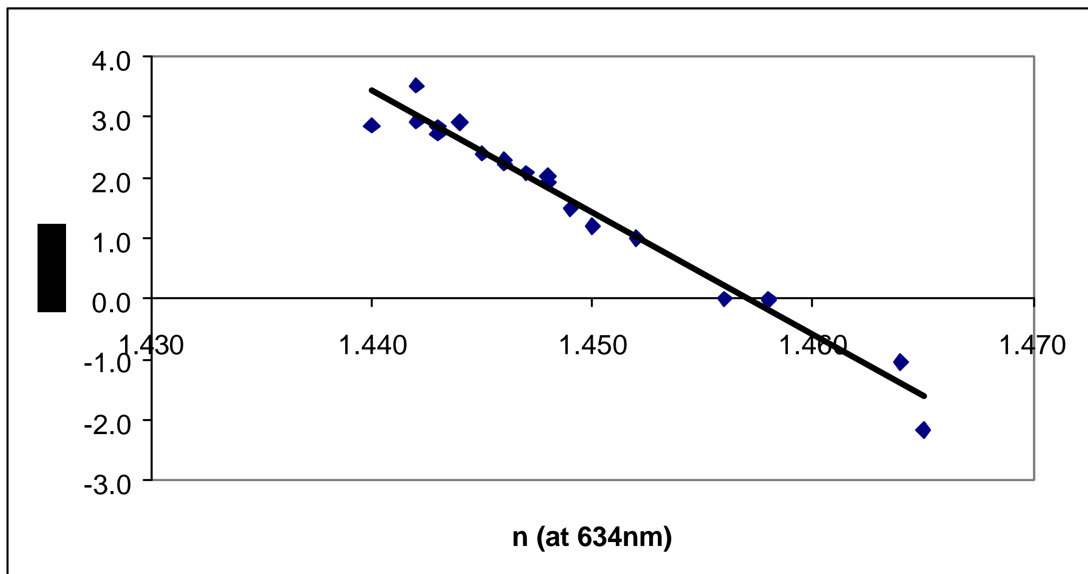


Figure 3 Plot of SE void fraction vs refractive index at 633nm

Wafer ID	Thickness SiO2 (nm)	% void	n (at 634nm)
K198 (AR wafer)	1.9	0.0	
thermal oxide	95.9	-2.2	1.465
75% process	99.5	3.5	1.442
DOE1 iterative process	125.4	2.2	1.446
DOE2 as deposited	87.4	2.9	1.440
95% Conformal Process	150.9	2.0	1.448
DOE2 anneal 850oC	95.1	2.9	1.442
75% process annealed at 850oC	95.2	2.7	1.443
95% process annealed at 850oC	142.3	2.1	1.447
95% process RTA 900oC	145.5	2.9	1.444
75% process annealed at 950oC	93.3	1.5	1.449
95% process annealed at 950oC	138.6	1.0	1.452
75% process RTA 1000oC	95.4	2.4	1.445
95% process RTA 1000oC	144.0	1.9	1.448
DOE1 annealed at 1050oC	116.9	0.0	1.456
DOE2 annealed 1050oC	99.5	-1.1	1.464
Ozone TEOS annealed at 1050oC	184.7	0.0	1.450
75% process annealed at 1050oC	92.0	1.2	1.450
95% process annealed at 1050oC	137.9	0.0	1.458
95% process annealed at 1100oC	138.0	0.0	1.458

Table 1 Summary of SE results from TEOS oxide layers.

3.2 X-ray photoelectron spectroscopy (XPS)

The XPS analysis provides information on the Si-O bonding energies and all films indicated similar qualitites, as shown in Figure 4. The main peaks observed were the O1s, Si2p, Si2s and C1s photoemission peaks at binding energies of 100, 150, 285 and 533eV respectively, with O and C Auger KLL peaks at 740 and 990eV. No signals from any other elements were detected. Table 2 summarizes the quantitative results derived from the spectra. O:Si stoichimetries were measured to be 2.0 within error in all cases except DOE2, which appeared slightly O-rich. This is expected if significant hydroxyl group concentrations are present and may be due to higher leak rates in the system. Si-O bonding state information may be obtained by the energy separation between O1s and Si2p peaks. The O1s peak generally has smaller chemical shifts than the Si2p peak, and the slight downward shift in the Si2p binding energy after annealing, could suggest a reduced degree of polarity in the layer bonding. The %Carbon detected was similar in all cases and due to surface layers.

Sample	TEOS conditions	anneal	O : Si		C	
			ratio	pk sepn(eV)	%	ML
K200	Thermal oxide	-	1.94	429.6	2.4	0.6
K134	75% process	None	2.03	429.5	6.1	1.5
K125	75% process	1050°C dens	1.97	429.6	3.8	0.9
K153	85% DOE1	None	2.00	430.6	5.9	1.4
K171	95% process	None	1.99	430.1	12.1	3.2
K163	95% process	1050°C dens	2.04	429.6	3.1	0.7
K190	DOE2	None	2.11	429.6	4.5	1.1

Table 2 Summary of XPS results

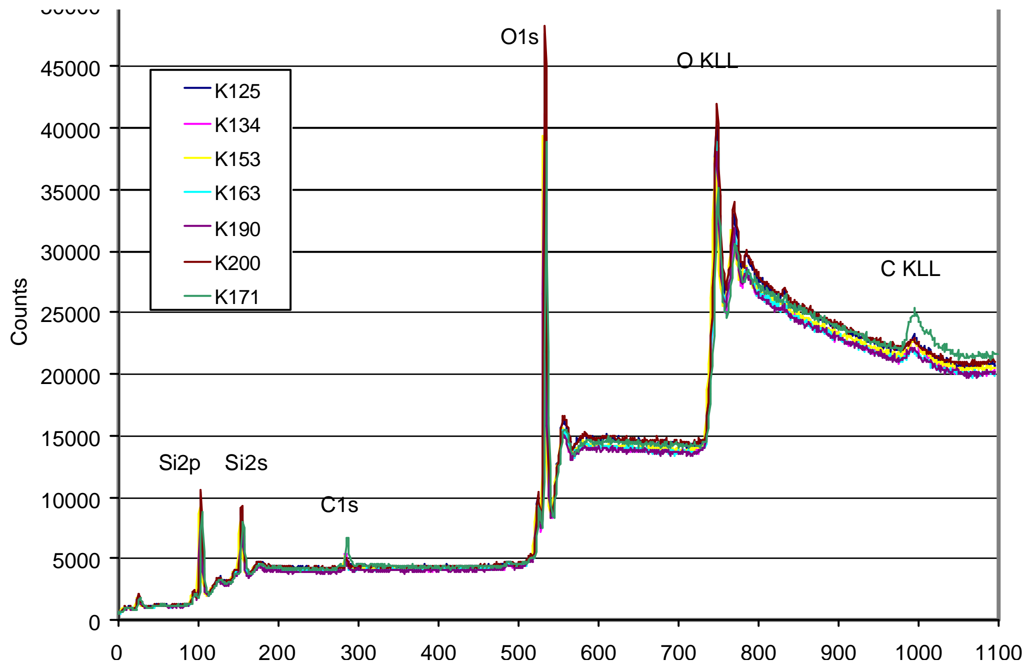


Figure 4 Wide scan XPS spectra from TEOS layers

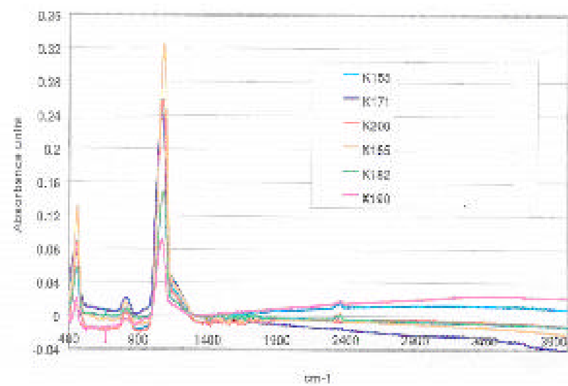
3.4 Infrared spectroscopy (IR).

Example full-range IR spectra are shown in Figure 5(a). The major peaks are those associated with Si-O stretching near 1070cm^{-1}, O-Si-O bending near 800cm^{-1} and O-Si-O rocking near 450cm^{-1}. A weak peak, sometimes negative, near 670cm^{-1}, is the subtracted substrate Si signal. Figures 5(b)-5(d) show three regions of the spectrum in more detail. In the high wave number range, a very weak feature is seen in four of the layers, comprising a broad band at 3200-3600cm^{-1} with a separate peak near 3650cm^{-1}. These are characteristic of H-bonded and free hydroxyl groups respectively, and are detected above background (~0.001 absorbance unit) only in the four as-deposited TEOS layers. This confirms that all the annealing treatments carried out are effective in layer dehydration. These four layers also give a weak additional shoulder near 950cm^{-1} which may be the –OH group bending vibration. The peak absorbance is related to the as deposited thickness.

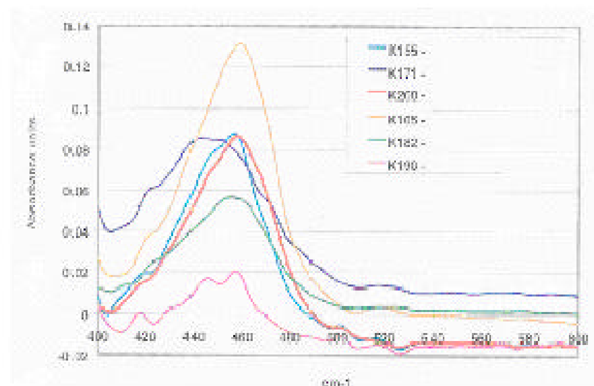
The broad band at 415-475cm^{-1} contains a considerable level of fine-structure related to detailed structural differences in the SiO_2 network. This additional structure is once again particularly noticeable in the four as-deposited layers. At least three discrete components – near 420, 445 and 455cm^{-1} – are evident in some cases and indicate less ordered structures. Table 5 gives the in Si-O stretching peak absorbances for the various films and Table 3 summarizes the anneal treatments of the DOE iterations.

Process parameters	Without Dense	Dense at 850°C	RTA at 900°C	Dense at 950°C	RTA at 1000°C	Dense at 1050°C	Dense at 1100°C
75% TEOS process	K134	K117	-	K121	K132	K125	-
85% DOE iteration 1	K153	-	-	-	-	K144	-
95% process	K171	K155	K167	K159	K169	K163	K165
DOE iteration 2	K190	K173	-	-	-	K182	-

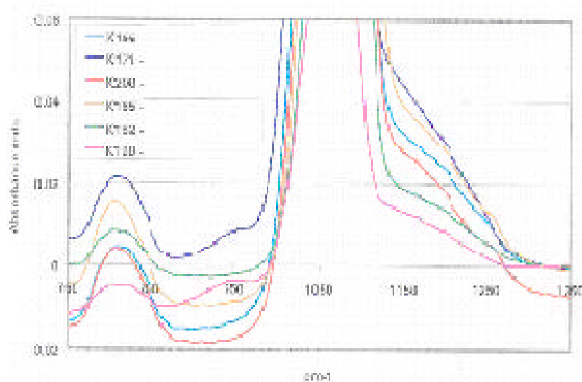
Table 3 Summary of 1kA TEOS layer deposition and treatment conditions



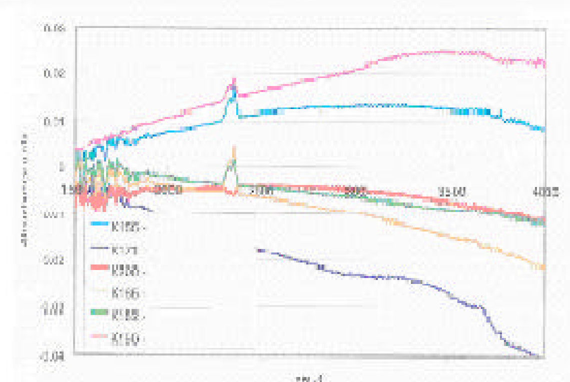
5(a)



5(b)



5(c)



5(d)

Figure 5 (a) Silicon subtracted 400-4000cm-1 IR absorbance spectra from six TEOS layers (b) Silicon subtracted IR absorbance spectra of the Si-O-Si rocking peak 5(c) Silicon subtracted IR absorbance spectra of the Si-O stretch and bend regions 5(d) Silicon subtracted IR absorbance spectra covering the –OH stretch region

Figure 6 depicts the FTIR transmission spectra for all the investigated TEOS films showing the main SI-O stretch and bend regions. There are signs of hydroxyl groups at the 950cm-1 frequency and a peak frequency lower than thermal TEOS. This is interpreted as a more disorganized and oxygen rich lattice. Figure 7 graphs TEOS films densified at 1050oC including Ozone TEOS. There are no obvious sign of hydroxyl groups, as seen in Figure 6. The main Si-O peak is higher in frequency than the thermal oxide peak indicating a frequency shift as the TEOS lattice rearranges under thermal annealing. This indicates a resolving of the silicon dioxide lattice to a more SiO_4 structure. The Ozone TEOS sample indicates a wider Si-O peak region indicating a different ratio lattice formation than the LPCVD TEOS films.

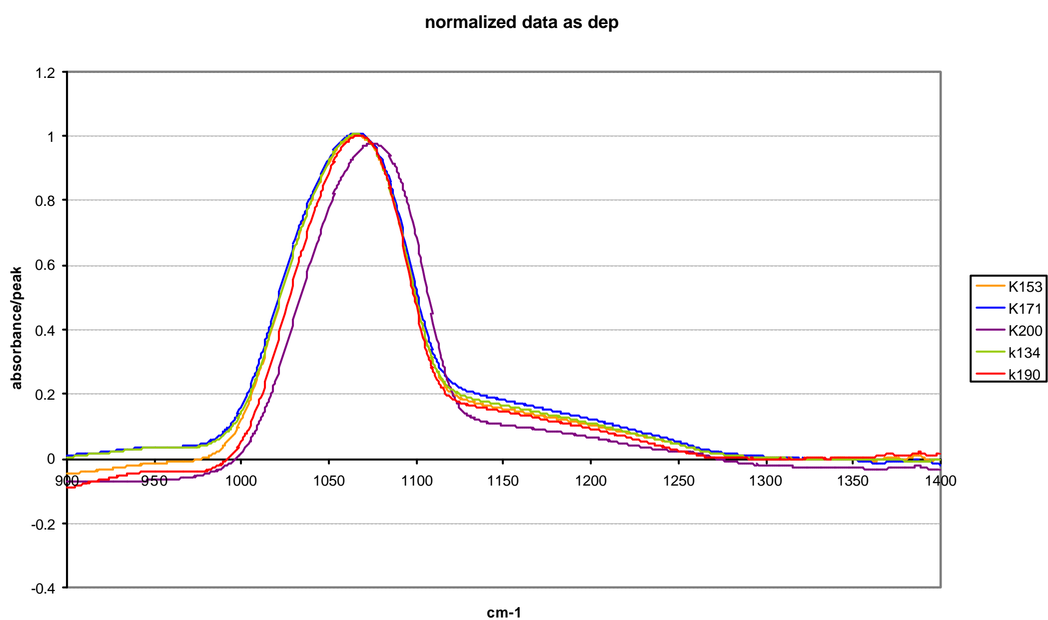


Figure 6 FTIR transmission spectra for TEOS films as deposited

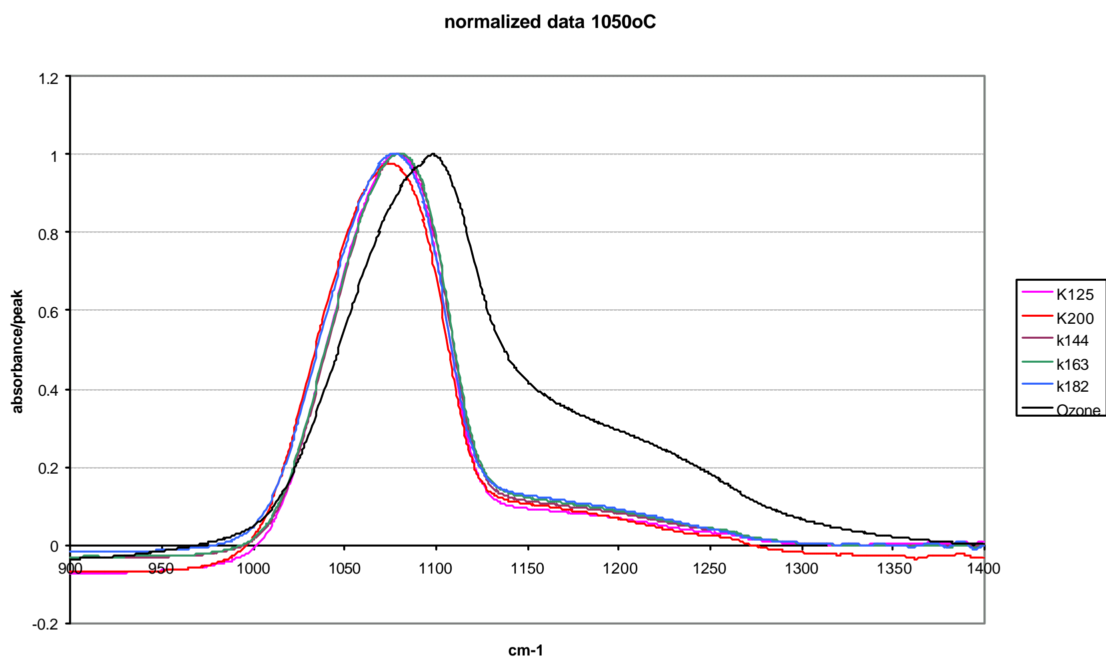


Figure 7 FTIR transmission spectra for TEOS films annealed at 1050oC

Wafer ID	Si-O peak posn (cm-1)	Si-O peak abs. (abs units)	OH peak abs (abs units) at 950cm^{-1}
K200	1073	0.264	
K190	1066	0.091	0.002
K182	1076	0.148	
K173	1071	0.123	
K171	1065	0.240	0.004
K169	1073	0.297	
K167	1071	0.284	
K165	1079	0.325	
K163	1078	0.314	
K159	1077	0.296	
K155	1072	0.258	
K153	1065	0.212	0.004
K144	1078	0.264	
K134	1062	0.168	0.003
K132	1070	0.187	
K125	1078	0.194	
K121	1074	0.194	
K117	1071	0.196	

Table 4 Summary of IR results : SiO and OH peak positions & intensities

Figure 7 indicates the relationship between anneal temperature and O-H peak intensity and shows that a 1050oC anneal is the optimal densification temperature.

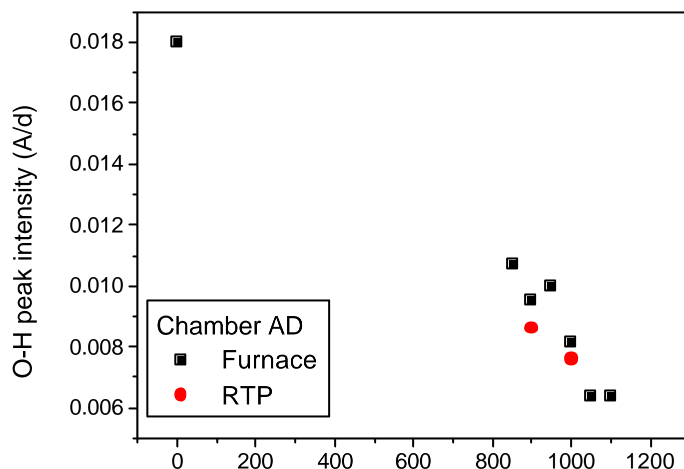


Figure 7 The dependence of O-H peak intensity in the region 3000-3500cm^{-1} on annealing temperature

Figures 8 show correlations between the IR Si-O stretch peak position, vs the SE void fraction respectively. The movement of the IR peak indicates the ratio of Si-O bonds in the structure and the void fraction relates to the film density. The graph indicates that the optimized process is denser than the other DOE films produced and shows a curve of IR Si-O peak position as the lattice is formed.

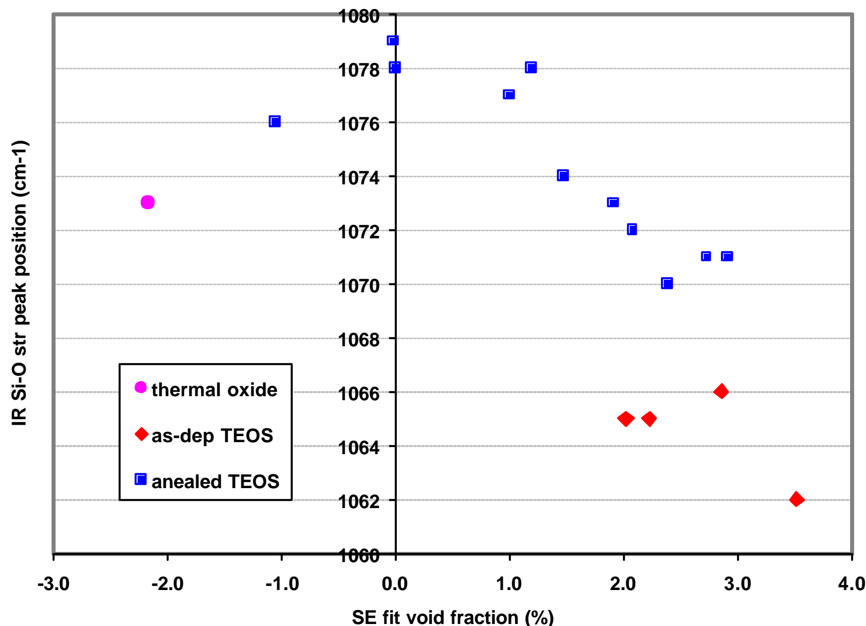


Figure 8 Plot of Si-O stretch IR peak position vs SE fit void fraction

3.5 Electrical Results

Table 5 indicates the breakdown voltage achieved by a variety of TEOS films and shows that the optimized process does not suffer from a degradation in breakdown voltage after sufficient annealing but as deposited values are lower.

Process condition	Breakdown Current (uA)	Breakdown Voltage (MV/cm)
75% process annealed at 900oC	10.0	8.7
75% process annealed at 1000oC	1.17	8.6
85% annealed at 1000oC	7.08	8.5
85% process annealed at 900oC	1.4	7.5
95% process annealed at 900oC	1.01	6.3
95% process annealed at 1000oC	2.3	8.4

Table 5 Electrical parameters for deposited TEOS films.

4. DISCUSSION

Without discussing the individual process characteristics it is difficult to fully explain the attributes investigated. The data provided indicates that the current LPCVD TEOS process available at Analog Devices Belfast is able to perform deep trench refill without producing voids. Voids in trenches are something which will frustrate many MEMs designers when they attempt to realize production of their designs. The optimized film produced has a reduced

uniformity, still within acceptable production limits, and has no other drawbacks compared to conventional LPCVD processes. For facilities with an LPCVD TEOS system, and are cost conscious, careful process optimi zation will allow TEOS films to be produced of similar quality and characteristics without the need for new equipment. Ozone TEOS may be chosen for new facilities or facilities without an LPCVD TEOS system and it does have a superior added particle performance. The main aim of optimizing the TEOS layer was to produce highly conformal layers, and the 95% conformal process exceeded expectations. While the initial work has indicated some variation in the conformality results, further process control is expected to lower the variations. The SEM images show a highly conformal layer with no bread-loafing in all cases. The variation in conformality could actually be due to measurement differences caused by charging at the edge of the film.

Breakdown voltage is very important for dielectrics and is dependent on the densification process. As deposited TEOS films contain a large ratio of hydroxyl groups which are removed after annealing at 850oC, and subsequent annealing serves to restructure the silicon dioxide lattice. A temperature of 1000oC or more is required to reach the optimal breakdown voltage. The breakdown voltages of all films are similar after annealing, while the as deposited films show a correlation with deposition temperature. The ellipsometry results indicate that we have achieved a process with a lower percentage void fraction that existing processes and conversely a denser film. The film also has slightly less stress (1-2MPa versus 2-3MPa) after densification. The ellipsometry results indicated a similarity in density between the optimized process and Ozone TEOS. The XPS results indicate similar structures between the films and no large proponent of Carbon or hydrogen in the films. Ozone TEOS is reported to have larger amounts of Carbon mainly due to the lower deposition temperature[(3)].

The FTIR results provide data on the crystalline structure on each of the DOE films. The aim of TEOS development is to produce a film as close to thermal oxide in structure as possible. The silicon dioxide films display a shoulder after the main peak at 1050cm-1. This indicates a greater proportion of Si_3O_2 and Si_2O_3 arrangements in the lattice. The as deposited films show a higher ratio of shoulder peak to the main peak and the shoulder reduces after annealing to a similar 10% ratio in all cases except Ozone TEOS which has a 30% ratio after densification. This appears to be correlated to the low deposition temperature of Ozone TEOS. Another indicator of TEOS quality is the movement of the Si-O peak from as deposited to after annealing as shown in Table 5. The crystalline structure shifts from a higher ratio of oxygen to silicon lattice structure to a more defined and high ratio of silicon lattice. When plotted against void fraction and related to deposition temperature the Silicon dioxide lattice formation can be optimized.

5. CONCLUSION

A process has been developed which allows highly conformal oxide refills of deep trenches with a 89° to 90° taper. It circumvents the need for tulip etches and two step etch processes or two step oxide deposition processes. SEM imaging of repeated production runs give within-wafer and wafer to wafer uniformity data of 3% which meets production requirements. Run to run variation is 5% which is less than standard processes optimized for uniformity at 3%, but still provides a controllable process. Variation in conformality has been measured at 89-100% across the furnace and along the trench depth up to 50um with a 100wafer load. The breakdown voltage is similar to conventional TEOS processes and physical characterization indicates a similar crystalline structure to other TEOS films. This process is currently used in production for oxide refills in MEMS applications. It has also been used to improve the breakdown voltage performance of high voltage products that have slip dislocations due to buried layer implants.

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REFERENCES

(1) TEOS deposition of Glass, A. Hochberg. Schumacher.
(2) The step coverage of undoped and phosphorous-doped SiO2, R.M. Levin and K. Evans-Lutterodt
(3) Electrical properties of bulk silicon dioxide formed by TEOS-ozone CVD, Hyo-Uk Kim and Shi-Woo Rhee.
(4) Control of Crystalline defects in trench isolated thick film SOI for high voltage smart power Ics, X. Cao, D. Nicholson, W.A. Nevin, J. Knopke