

**ROCHESTER INSTITUTE OF TECHNOLOGY
MICROELECTRONIC ENGINEERING**

Low Pressure Chemical Vapor Deposition

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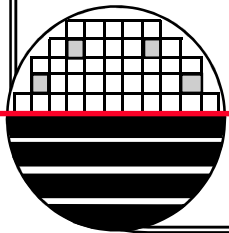
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MicroE webpage: <http://www.microe.rit.edu>

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Microelectronic Engineering*

11-26-2013 LPCVD_Recipes.ppt



OUTLINE

Introduction

LPCVD Nitride

Stoichiometric

Low Stress – Silicon Rich

Stress in Nitride Films

LPCVD Poly

Poly for CMOS (6000Å)

Poly for MEMS (2µm High Dep Rate)

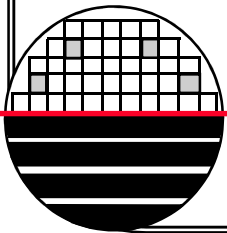
Stress in Poly Films

Doping Poly

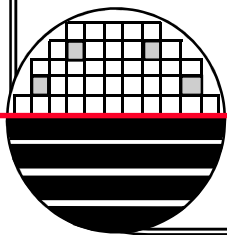
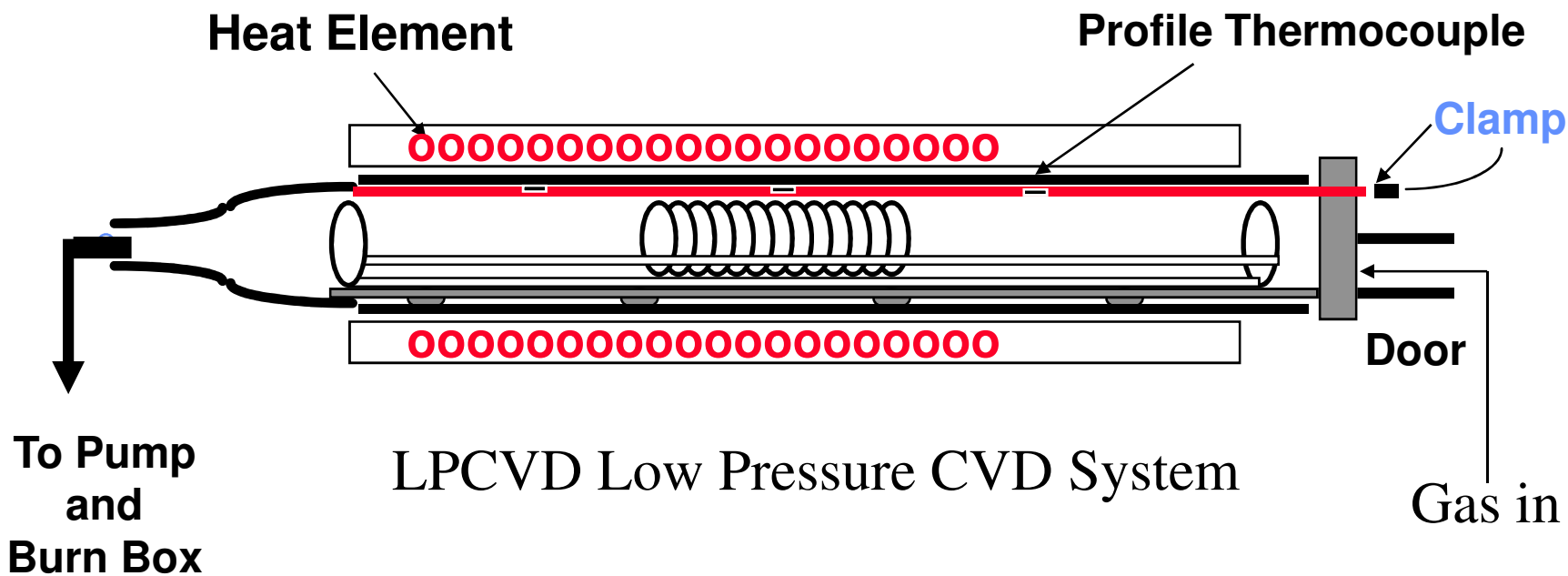
LPCVD Oxide (LTO)

Uniformity

LPCVD Parylene



**LOW PRESSURE CHEMICAL VAPOR DEPOSITION
(LPCVD)**



RIT LPCVD TOOLS

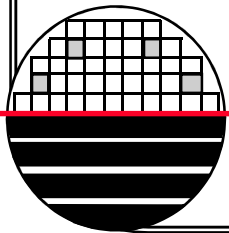


6" LPCVD

4" LPCVD

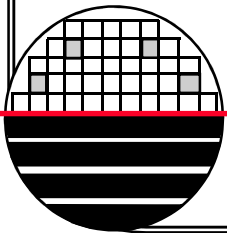
Top Tube for LTO

Bottom Tube for Poly and Nitride



BURN BOX

The correct name is controlled combustion/reaction system. Exhaust gas exits through the “burn box” which is heated to 850 °C. At this temperature the exhaust gas reacts with air and any material that can burn is burned under controlled conditions within the “burn box”



6" LPCVD STOCIOMETRIC NITRIDE

Silicon Nitride (Si₃N₄) (normal - stociometric):

Temperature = 810 °C Flat from (door to pump)

Pressure = 400 mTorr $3\text{SiH}_2\text{Cl}_2 + 4\text{NH}_3 = \text{Si}_3\text{N}_4 + 9\text{H}_2 + 3\text{Cl}_2$

Dichlorosilane (SiH₂Cl₂) Flow = 60 sccm

Ammonia (NH₃) Flow = 150 sccm

Rate = 60 Å/min +/- 10 Å/min

non uniformity of ~ 10%

At 300 mTorr with same conditions as above

Rate = 55 Å/min +/- 10%

non uniformity ~ 3%

Silicon Nitride (Si₃N₄) (low - stress)

Temperature = 800 °C Flat

Pressure =

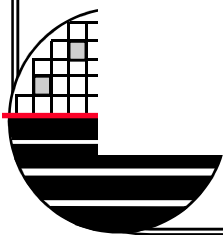
Dichlorosilane = 200

Ammonia = 20

Rate =

non uniformity =

Tough on pump and burn box, lots of HCL and Cl byproducts



4" LPCVD STOCIOMETRIC NITRIDE

Silicon Nitride (Si₃N₄) (normal - stociometric):

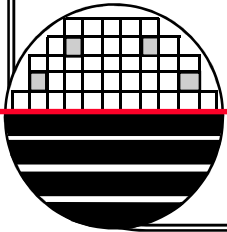
Temperature = 790-800-810 °C Ramp from (door to pump)

Pressure = 375 mTorr $3\text{SiH}_2\text{Cl}_2 + 4\text{NH}_3 = \text{Si}_3\text{N}_4 + 9\text{H}_2 + 3\text{Cl}_2$

Dichlorosilane (SiH₂Cl₂) Flow = 60 sccm

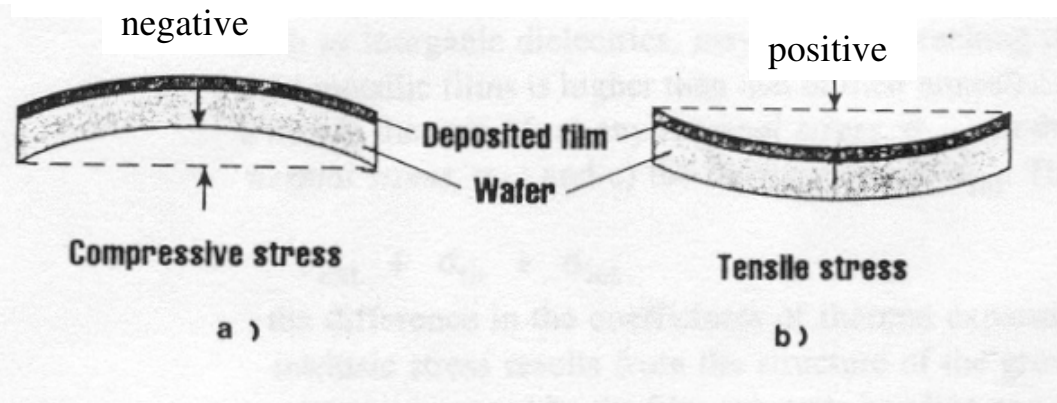
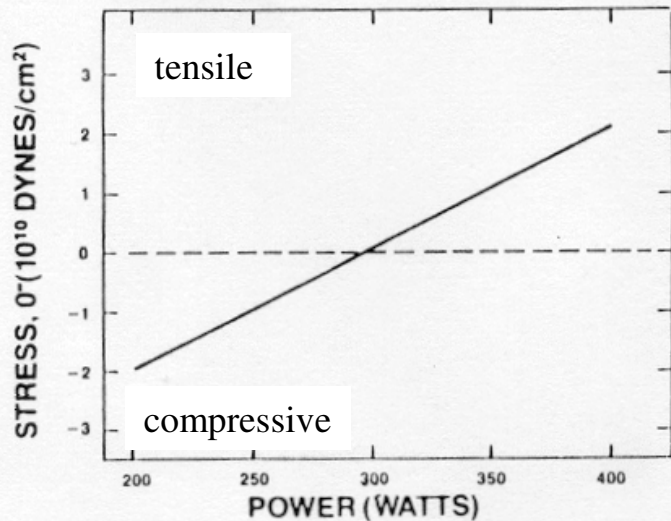
Ammonia (NH₃) Flow = 150 sccm

Rate = 60 Å/min +/- 10 Å/min



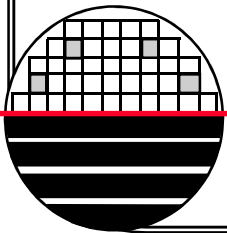
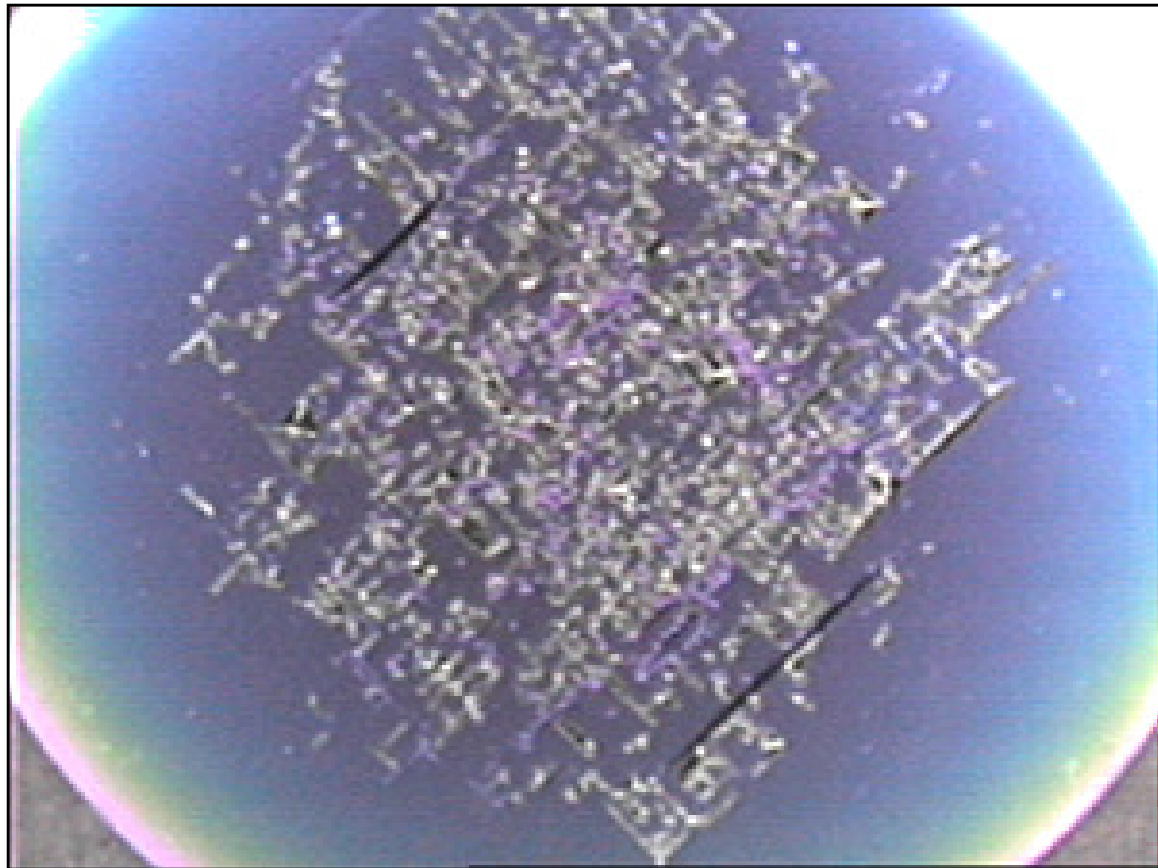
STRESS IN SILICON NITRIDE FILMS

Compressively stressed films would like to expand parallel to the substrate surface, and in the extreme, films in compressive stress will buckle up on the substrate. Films in tensile stress, on the other hand, would like to contract parallel to the substrate, and may crack if their elastic limits are exceeded. Stresses can be negative or positive or near zero depending on many parameters.



STRESS IN NITRIDE FILMS

Stress in an 8000 Å Nitride Film causing fracture



LOW STRESS SILICON RICH Si3N4

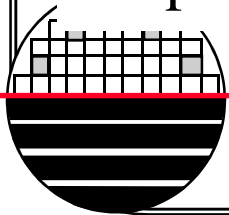
ADE Measured stress for various Ammonia:Dichlorosilane Flow Ratios

Flow	Stress x E 9 dynes/cm ²
10:1	+14.63
5:1	+14.81
2.5:1	+12.47 *
1:1	+10.13
1:2.5	+7.79
1:5	+3
1:10	0

* standard recipe

Stress; $\sigma = (E/(6(1-\nu))) * (D^2/(rt))$
 where E is Youngs modulus,
 ν is Poissons ratio,
 D and t are substrate and film thickness
 r is radius of curvature (+ for tensile)

T.H Wu, “Stress in PSG and Nitride Films as Related to Film Properties and Annealing”, Solid State Technology, p 65-71, May ‘92



MEASUREMENT OF STRESS IN Si₃N₄

Kenneth L. Way, Jr. did his senior project on stress in silicon nitride films as a function of the ratio of ammonia to dichlorosilane. Samples were coated with various flows and stress was measured at ADE corporation. The silicon nitride was etched off of the backside of the wafer so that the stress curvature was due to the layer on the front side only. Dr. Lane said the nitride runs at 1:10 (ammonia:dichlorosilane) ratios are rough on the pumping system.

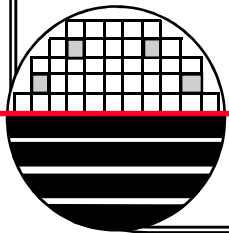
Compressive Stress



Tensile Stress



Dr. Grande sent samples to Kodak for stress measurement. He found stress of +900 MPa Tensile for the standard Nitride recipe for 1500 A thickness, 1-29-2000



6" LPCVD LOW STRESS SILICON NITRIDE

Silicon Nitride (Si₃N₄) (low - stress)

Temperature = 800 °C Flat

Pressure =

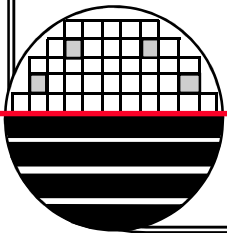
Dichlorosilane = 200

Ammonia = 20

Rate =

non uniformity =

Tough on pump and burn box, lots of HCL and Cl byproducts, we do not run this process very often.



4" LPCVD LOW STRESS SILICON NITRIDE

Silicon Nitride (Si₃N₄) (low stress – silicon rich):

Temperature = 790-800-810 °C Ramp from (door to pump)

Pressure = 650 mTorr $3\text{SiH}_2\text{Cl}_2 + 4\text{NH}_3 = \text{Si}_3\text{N}_4 + 9\text{H}_2 + 3\text{Cl}_2$

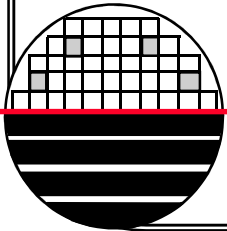
Dichlorosilane (SiH₂Cl₂) Flow = 200 sccm

Ammonia (NH₃) Flow = 20 sccm

Rate = 80 Å/min +/- 10 Å/min

Films can be deposited up to about 5000 Å directly on silicon before the stress is so large that the film fractures (Dr. Lane, Dr. Fuller). Pad oxide under the nitride film and special silicon rich nitride films may allow nitride film thickness over 5000 Å.

9-6-96 Dr. Fuller did a 100 min deposition giving 8100Å (center) to 8800Å (edge) nitride thickness which did not fracture due to stress. It is a definite improvement.



6" LPCVD POLYSILICON FOR CMOS

Polysilicon Deposition:

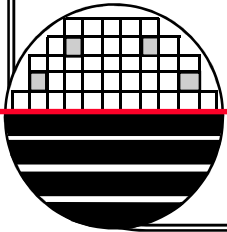
Temperature = 610 °C

Pressure = 300 mTorr

Gas = Silane (SiH₄)

Flow = 90 sccm

Rate = 64 Å/min

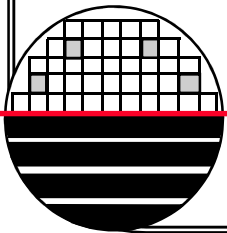


6" LPCVD POLYSILICON FOR MEMS

High Dep. Rate Polysilicon Deposition:

Temperature = 650 °C $\text{SiH}_4 = \text{Si} + 2\text{H}_2$
Pressure = 300 mTorr
Gas = Silane (SiH_4)
Flow = 400 sccm
Rate = 200 Å/min (~ 3 times the normal dep. rate at 610 °C)

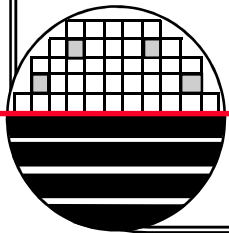
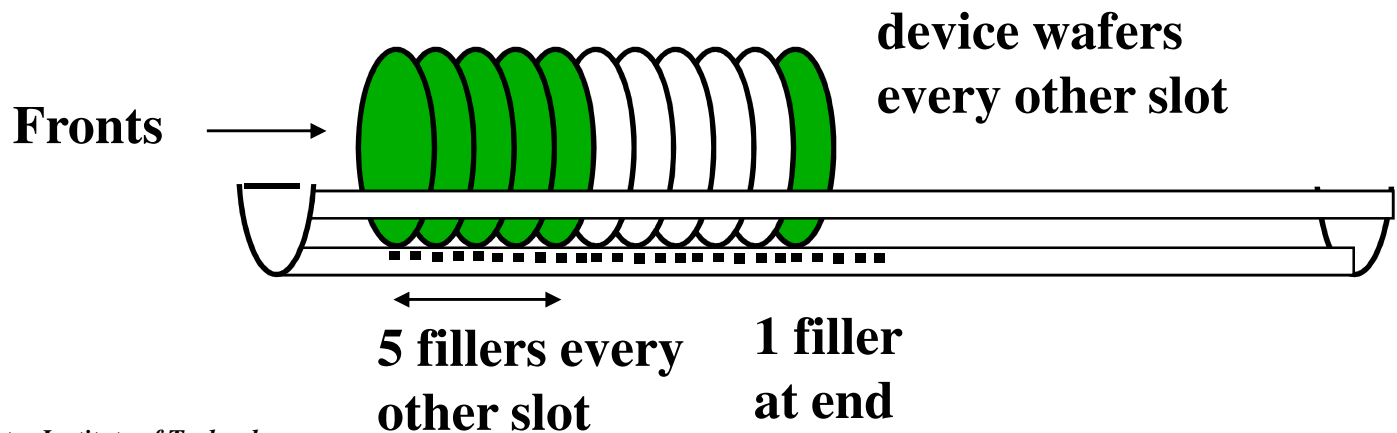
Thicker films have larger grain size (grain size ~ equal to film thickness) and thus a rougher appearance.



4" LPCVD POLYSILICON FOR CMOS

Polysilicon Deposition:

Temperature = 610 °C
Pressure = 300 mTorr
Gas = Silane (SiH₄)
Flow = 90 sccm
Rate = 77 Å/min

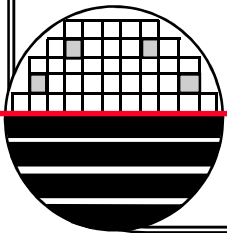


4" LPCVD POLYSILICON FOR MEMS

High Dep. Rate Polysilicon Deposition:

Temperature = 650 °C $\text{SiH}_4 = \text{Si} + 2\text{H}_2$
Pressure = 300 mTorr
Gas = Silane (SiH_4)
Flow = 90 sccm
Rate = 235 Å/min (~ 3 times the normal dep. rate at 610 °C)

Thicker films have larger grain size (grain size ~ equal to film thickness) and thus a rougher appearance.



DOPING POLYSILICON FROM SPIN-ON DOPANTS

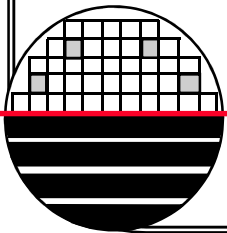
When using poly as a conductor in integrated circuits it is desirable to have low resistivity. Doping at 1000 °C for 20 min using Emulsitone Co., 19 Leslie Court, Whippany, NJ 07981 Tel (201)386-0053; Emitter Diffusion Source N250 spin-on dopant gives 10-15 ohm/sq sheet resistance for 0.75 um thick poly. (The Allied Signal Inc., 1090 South Milpitas Boulevard, Milpitas, CA 95035, Tel (408)946-2411, Accuspin P-854 dopant gives higher resistivity in the range of 100 ohm/sq.)

There is no problem unless one is concerned with the possibility of the dopant going through a thin gate oxide and affecting the underlying substrate. In this case the doping needs to be adequate however the subsequent high temperature steps each drive the dopant further through the gate oxide. CV measurements can be used to see if the dopant gets through to the substrate. For n-type dopant and p-type substrate one would expect a shift to the left if the dopant goes through the gate. We found that doping at 1000 C for 10 min soak, using N250 spin on source, followed by 60 min in nitrogen at 1000 C did not cause a shift to the left in the CV plot. (actually shifted right similar to anneal or sinter) We did not test the breakdown voltage or effect of gate oxide doping on breakdown voltage. We did not try additional time at 1000 C in Nitrogen looking for shift to left in CV plot.

DOPING POLYSILICON BY ION IMPLANT

In the advanced CMOS process we want N+ poly on the NMOSFET and P+ poly on the PMOSFET. So the poly is deposited undoped and during D/S implant the poly is also doped.

In the submicron CMOS process we dope the poly after deposition n+ for both transistors. We use a dose of $2E16$ which is the highest dose that we typically do at RIT. Since there is no resist on the wafer we do not have to consider heating effects during implant. The current is set as high as possible, often 500uA or more.

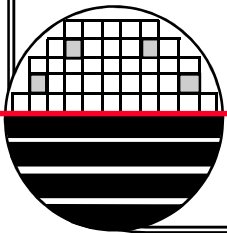


DOPING POLYSILICON BY ION IMPLANT

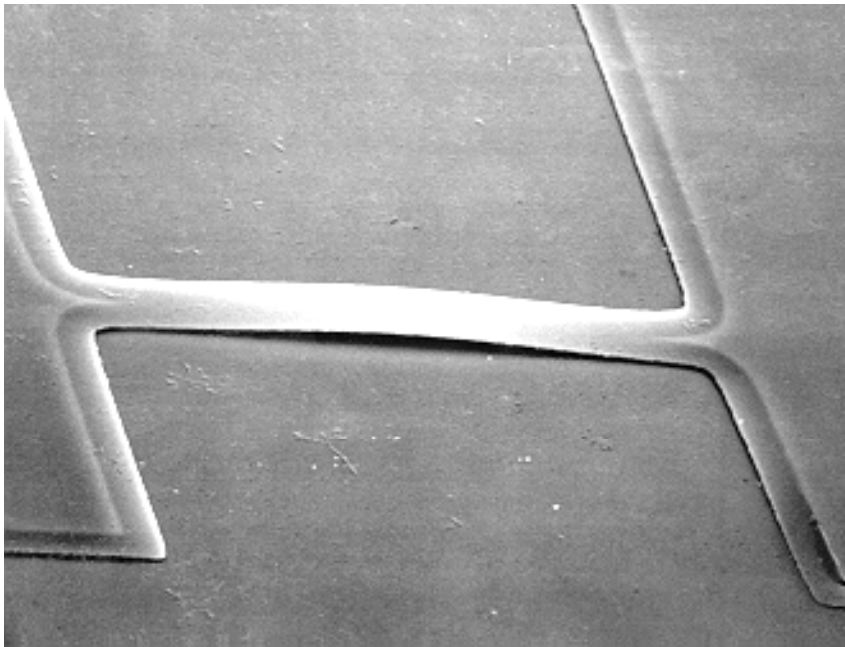
In some analog CMOS processes we want high sheet resistance poly resistors for voltage dividers, etc. The measured resistor data below shows sheet resistance versus Boron (B11) dose for a 3500Å poly layer after 30 min. 1000 °C anneal.

$R=1/\text{slope}$; $R_{\text{hos}}=R / \# \text{sq}$; $R_{\text{ho}}=R_{\text{hos}} \times \text{thickness (3500\AA)}$; Dose=implanter setting

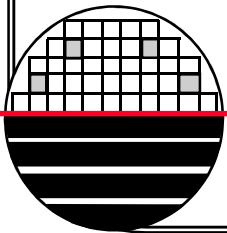
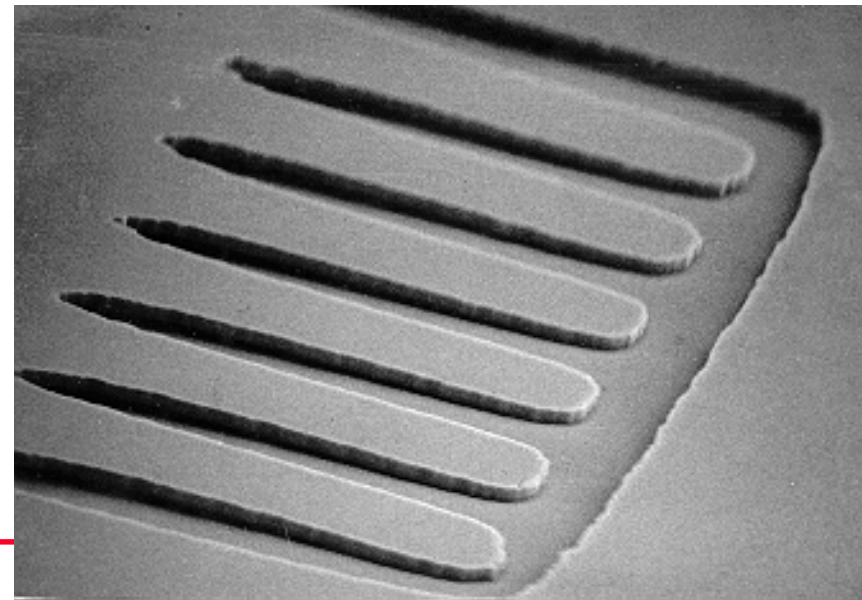
R wafer 4 = 106 G ; $R_{\text{hos}} = 2.94 \text{ Gohm/sq}$; $R_{\text{ho}} = 103\text{K ohm-cm}$; Dose=1E12 cm⁻²
 R wafer 3 = 339 G ; $R_{\text{hos}} = 9.42 \text{ Gohm/sq}$; $R_{\text{ho}} = 330\text{K ohm-cm}$; Dose = 3E11
 R wafer 2 = 943 G ; $R_{\text{hos}} = 26.2 \text{ Gohm/sq}$; $R_{\text{ho}} = 917\text{K ohm-cm}$; Dose = 6E11
 R wafer 1 = 1104 G; $R_{\text{hos}} = 30.7 \text{ Gohm/sq}$; $R_{\text{ho}} = 1075\text{K ohm-cm}$; Dose = 1E11



STRESS IN POLY FILMS

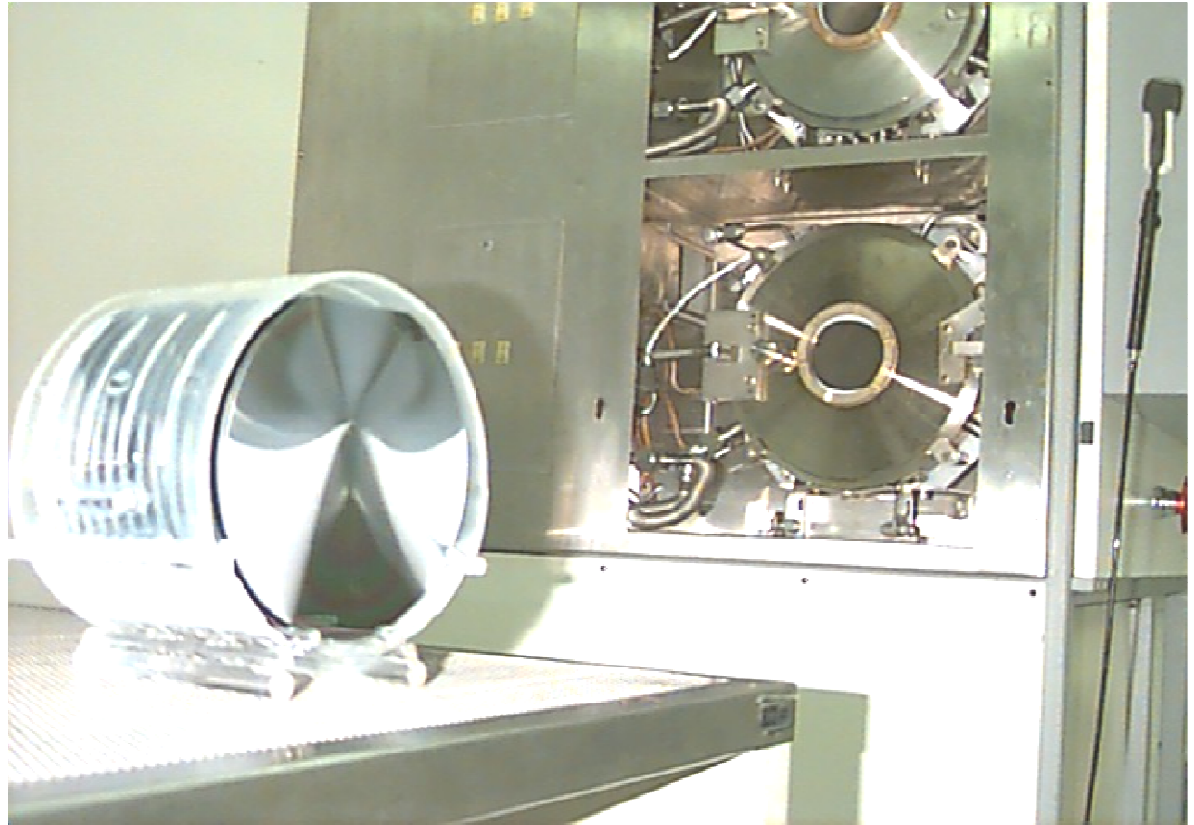


Stress in poly films can cause buckling and bending of beams and cantilever structures. When doping poly after deposition the high temperatures (1000 C) will anneal stress. Undoped poly structures require an anneal.

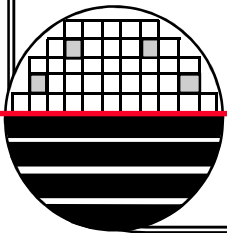


LOW TEMPERATURE OXIDE (LTO)

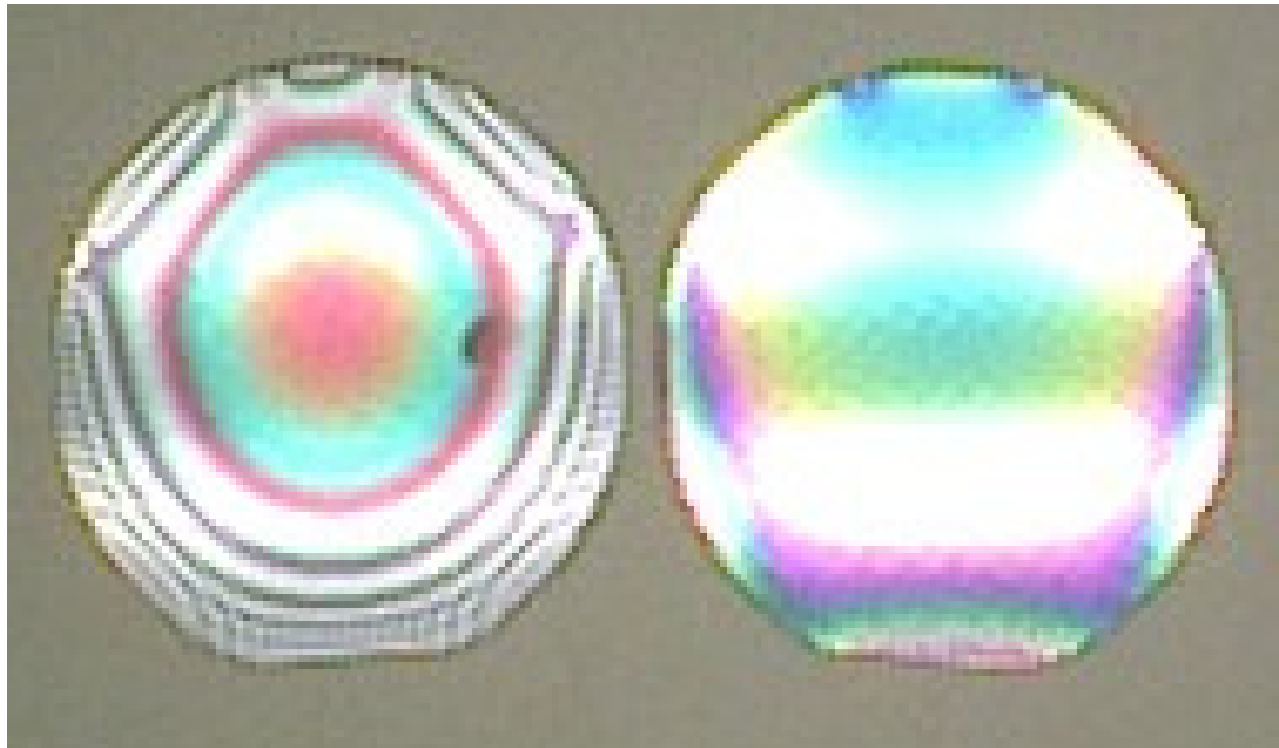
Wafers are loaded back to back in caged boat. The boat is filled with dummy wafer to total 25 wafers. Monitor wafer is placed in the middle. Injector tubes direct the gas (SiH_4 and O_2) directly under the middle of the caged boat.



Caged Boat



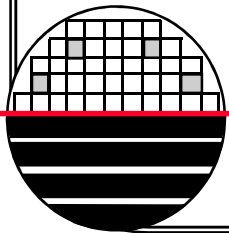
IMPROVED UNIFORMITY OF LTO WITH CAGED BOAT



In 4" LPCVD

Using 6" LPCVD
Caged Boat and Injectors

*Rochester Institute of Technology
Microelectronic Engineering*

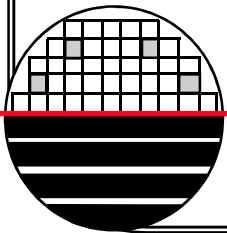


6" LTO RECIPE

Low Temperature Silicon Oxide:

Temperature = 400 °C $\text{SiH}_4 + \text{O}_2 = \text{SiO}_2 + 2\text{H}_2$
Pressure = 250 mTorr
Silane (SiH_4) Flow = 40 sccm
Oxygen (O_2) Flow 48 sccm
Rate = 70 Å/min +/- 10 Å/min

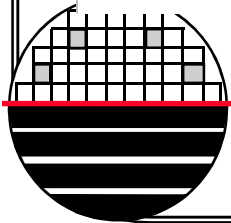
7-26-00 LTO @425 °C gave deposition rate of 113 Å/min



OXIDE THICKNESS COLOR CHART

Thickness	Color
500	Tan
700	Brown
1000	Dark Violet - Red Violet
1200	Royal Blue Blue
1500	Light Blue - Metallic Blue
1700	Metallic - very light Yellow Green
2000	Light Gold or Yellow - Slightly Metallic
2200	Gold with slight Yellow Orange
2500	Orange - Melon
2700	Red Violet
3000	Blue - Violet Blue
3100	Blue Blue
3200	Blue - Blue Green
3400	Light Green
3500	Green - Yellow Green
3600	Yellow Green
3700	Yellow
3900	Light Orange
4100	Carnation Pink
4200	Violet Red
4400	Red Violet
4600	Violet
4700	Blue Violet

Thickness	Color
4900	Blue Blue
5000	Blue Green
5200	Green
5400	Yellow Green
5600	GreenYellow
5700	Yellow - "Yellowish"(at times appears to be Lt gray or matel
5800	Light Orange or Yellow - Pink
6000	Carnation Pink
6300	Violet Red
6800	"British"(appears violet red, Blue Green, looks Blue
7200	Blue Green - Green
7700	"Yellowish"
8000	Orange
8200	Salmon
8500	Dull, Light Red Violet
8600	Violet
8700	Blue Violet
8900	Blue Blue
9200	Blue Green
9500	Dull Yellow Green
9700	Yellow - "Yellowish"
9900	Orange
10000	Carnation Pink



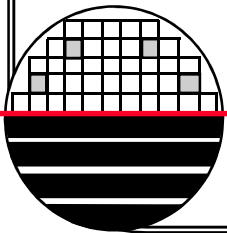
Nitride Thickness = (Oxide Thickness)(Oxide Index/Nitride Index)
 Eg. Yellow Nitride Thickness = (2000)(1.46/2.00) = 1460



NITRIDE MEASUREMENTS

Ellipsometer Program 7 does nitride on pad oxide
Nanospec Program 6 does nitride on pad oxide
Spectromap does nitride on pad oxide

You need to know the value of the pad oxide thickness.



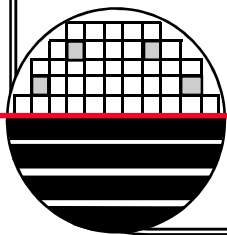
PARYLENE DEPOSITION SYSTEM

Approximately 1 gm of Parylene C gives $\sim 3000\text{\AA}$ film thickness, Deposit 5 wafers per run.

Discovered we need A174 Adhesion Promotor. Chemical name gamma-methacryloxypropyltrimethoxy silane. Spin coat straight from package at 2000 rpm. Bake on hot plate at 115 C for 1 min.



See: <http://www.scscookson.com/parylene/properties.cfm>



PARYENE MINI OPERATION MANUAL

Mini Operation Manual for the PDS-2010 – Parylene Deposition System
Revision 4-30-04

Dr. Lynn Fuller, Germain Fenger



Clean up Prior to Deposition:

1. Open the storage drawer under the white table. Contains Parylene powder, cleaning tools, microclean-90 spray cleaner, concentrate of microclean-90 and spare parts. (If necessary mix more microclean-90 by diluting with water a few caps full per spray bottle)
2. Clean around seal, on Bell Jar and Stage. Only clean the inside of Bell Jar and center on stage if there is visible peeling.
3. Spray microclean-90 on parts that have been cleaned and are exposed.
4. Verify that the small pressure sensor hole is clear of deposition, if it is not use a small hook tool to remove the deposition. Do not push the parylene residue further in the hole.
5. Clean the chiller (back right corner). Remove the chiller being careful of the tubes on the end of the chiller (inside of black cover) for they bend easily. Use a scour pad to remove the majority of the deposition, some deposition may require the use of a razor blade. Spray with micro-90 when done.
6. Remove Deposition Tube and verify that it is not clogged, or the hole that it goes into.

Set up for Deposition:

1. Open the front cabinet door. Unclamp and open the vaporizer door on the lower left. Remove the aluminum foil Dimer boat. If you need to make a new Dimer boat see page 18 of the manual.
2. One gram of Parylene type C from Specialty Coating Systems, 5707 West Minnesota St., Indianapolis, IN 46241, gives 1 micrometer of thickness. Measure out the desired amount of Parylene powder. Use the gram scale near the 4 pt probe. Lift off the glass cover, turn ON, set zero >0, set foil on top of scale, set tear T< (reading should go to zero), using the scoop put some powder on the foil to desired amount. Pour the measured amount of powder into the aluminum foil Dimer boat.
3. Place boat with powder in the vaporizer and close and clamp the door.
4. Place wafers in the designated boats (in storage cabinet).

Start System:

1. Card Swipe in.
2. Place wafers on stage, pull EMO, turn the switch labeled vaporizer on and verify that the boat does not rub on anything, if it is found to be rubbing adjust boat position, turn off vaporizer.
3. Place bell jar on stage.
4. Turn on vaporizer, and again verify that the boat is not rubbing, if it is rubbing remove bell jar and adjust.
5. While holding the chiller in place, turn right switch to vacuum, once under vacuum you may release the chiller.
6. Turn on the chiller, (left green switch)
7. Turn on furnace switch and vaporizer.
8. Once pressure reads below 30mTorr (base pressure) push the green button (right).

REFERENCES

1. Silicon Processing for the VLSI Era, Volume 1, 2nd Edition, Stanley Wolf, Richard Tauber, Lattice Press, 2000.
2. The Science and Engineering of Microelectronic Fabrication, Stephen A. Campbell, Oxford University Press, 1996.

