Low Pressure Chemical Vapor Deposition

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11-26-2013 LPCVD_Recipes.ppt
Introduction
LPCVD Nitride
  Stociometric
  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)

LPCVD Low Pressure CVD System

Heat Element

Profile Thermocouple

To Pump and Burn Box

Gas in

Clamp

Door

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RIT LPCVD TOOLS

6” LPCVD
Top Tube for LTO
Bottom Tube for Poly and Nitride

4” LPCVD
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
Silicon Nitride (Si3N4) (normal - stociometric):
  Temperature = 790-800-810 °C Ramp from (door to pump)
  Pressure = 375 mTorr
  $3\text{SiH}_2\text{Cl}_2 + 4\text{NH}_3 \rightarrow \text{Si}_3\text{N}_4 + 9\text{H}_2 + 3\text{Cl}_2$
  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 an 8000 A Nitride Film causing fracture
LOW STRESS SILICON RICH Si3N4

ADE Measured stress for various Ammonia:Dichlorosilane Flow Ratios

<table>
<thead>
<tr>
<th>Flow</th>
<th>Stress x E 9 dynes/cm²</th>
</tr>
</thead>
<tbody>
<tr>
<td>10:1</td>
<td>+14.63</td>
</tr>
<tr>
<td>5:1</td>
<td>+14.81</td>
</tr>
<tr>
<td>2.5:1</td>
<td>+12.47 *</td>
</tr>
<tr>
<td>1:1</td>
<td>+10.13</td>
</tr>
<tr>
<td>1:2.5</td>
<td>+7.79</td>
</tr>
<tr>
<td>1:5</td>
<td>+3</td>
</tr>
<tr>
<td>1:10</td>
<td>0</td>
</tr>
</tbody>
</table>

Stress; \( \sigma = \frac{E}{(6(1-v))} \frac{D^2}{rt} \)

where \( E \) is Youngs modulus,
\( v \) is Poissons ratio,
\( D \) and \( t \) are substrate and film thickness
\( r \) is radius of curvature (+ for tensile)

* standard recipe


10 dyne/cm² = 1 newton/m² = 1 Pascal
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. 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.

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.

10 dyne/cm² = 1 newton/m² = 1 Pascal
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
- 3SiH2Cl2 + 4NH3 = Si3N4 + 9H2 + 3Cl2
- 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.
Polysilicon Deposition:
- Temperature = 610 °C
- Pressure = 300 mTorr
- Gas = Silane (SiH4)
- Flow = 90 sccm
- Rate = 64 Å/min
High Dep. Rate Polysilicon Deposition:

Temperature = 650 °C       \[ \text{SiH}_4 = \text{Si} + 2\text{H}_2 \]
Pressure = 300 mTorr
Gas = Silane (SiH4)
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.
Polysilicon Deposition:
Temperature = 610 °C
Pressure = 300 mTorr
Gas = Silane (SiH4)
Flow = 90 sccm
Rate = 77 Å/min

SiH4 = Si + 2H2

device wafers

Fronts

5 fillers every other slot

1 filler at end

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High Dep. Rate Polysilicon Deposition:

Temperature = 650 °C  \( \text{SiH}_4 = \text{Si} + 2\text{H}_2 \)
Pressure = 300 mTorr
Gas = Silane (\( \text{SiH}_4 \))
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.
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.
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.

\[ R = \frac{1}{\text{slope}}; \quad \text{Rhos} = \frac{R}{\text{sqs}}; \quad \text{Rho} = \text{Rhos} \times \text{thickness} (3500Å); \quad \text{Dose} = \text{implanter setting} \]

- **R wafer 4 = 106 G**; \quad \text{Rhos} = 2.94 \text{ Gohm/sq}; \quad \text{Rho} = 103K \text{ ohm-cm}; \quad \text{Dose} = 1E12 \text{ cm}^{-2}
- **R wafer 3 = 339 G**; \quad \text{Rhos} = 9.42 \text{ Gohm/sq}; \quad \text{Rho} = 330K \text{ ohm-cm}; \quad \text{Dose} = 3E11
- **R wafer 2 = 943 G**; \quad \text{Rhos} = 26.2 \text{ Gohm/sq}; \quad \text{Rho} = 917K \text{ ohm-cm}; \quad \text{Dose} = 6E11
- **R wafer 1 = 1104 G**; \quad \text{Rhos} = 30.7 \text{ Gohm/sq}; \quad \text{Rho} = 1075K \text{ ohm-cm}; \quad \text{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 (SiH4 and O2) directly under the middle of the caged boat.
IMPROVED UNIFORMITY OF LTO WITH CAGED BOAT

In 4” LPCVD

Using 6” LPCVD

Caged Boat and Injectors
6” LTO RECIPE

Low Temperature Silicon Oxide:
- Temperature = 400 °C
- Pressure = 250 mTorr
- Silane (SiH4) Flow = 40 sccm
- Oxygen (O2) Flow = 48 sccm
- Rate = 70 Å/min +/- 10 Å/min

SiH4 + O2 = SiO2 + 2H2

7-26-00 LTO @425 °C gave deposition rate of 113 Å/min
# OXIDE THICKNESS COLOR CHART

<table>
<thead>
<tr>
<th>Thickness</th>
<th>Color</th>
</tr>
</thead>
<tbody>
<tr>
<td>500</td>
<td>Tan</td>
</tr>
<tr>
<td>700</td>
<td>Brown</td>
</tr>
<tr>
<td>1000</td>
<td>Dark Violet - Red Violet</td>
</tr>
<tr>
<td>1200</td>
<td>Royal Blue</td>
</tr>
<tr>
<td>1500</td>
<td>Light Blue - Metallic Blue</td>
</tr>
<tr>
<td>1700</td>
<td>Metallic - very light Yellow Green</td>
</tr>
<tr>
<td>2000</td>
<td>Light Gold or Yellow - Slightly Metallic</td>
</tr>
<tr>
<td>2200</td>
<td>Gold with slight Yellow Orange</td>
</tr>
<tr>
<td>2500</td>
<td>Orange - Melon</td>
</tr>
<tr>
<td>2700</td>
<td>Red Violet</td>
</tr>
<tr>
<td>3000</td>
<td>Blue - Violet Blue</td>
</tr>
<tr>
<td>3100</td>
<td>Blue</td>
</tr>
<tr>
<td>3200</td>
<td>Blue - Blue Green</td>
</tr>
<tr>
<td>3400</td>
<td>Light Green</td>
</tr>
<tr>
<td>3500</td>
<td>Green - Yellow Green</td>
</tr>
<tr>
<td>3600</td>
<td>Yellow Green</td>
</tr>
<tr>
<td>3700</td>
<td>Yellow</td>
</tr>
<tr>
<td>3900</td>
<td>Light Orange</td>
</tr>
<tr>
<td>4100</td>
<td>Carnation Pink</td>
</tr>
<tr>
<td>4200</td>
<td>Violet Red</td>
</tr>
<tr>
<td>4400</td>
<td>Red Violet</td>
</tr>
<tr>
<td>4600</td>
<td>Violet</td>
</tr>
<tr>
<td>4700</td>
<td>Blue Violet</td>
</tr>
<tr>
<td>4900</td>
<td>Blue</td>
</tr>
<tr>
<td>5000</td>
<td>Blue Green</td>
</tr>
<tr>
<td>5200</td>
<td>Green</td>
</tr>
<tr>
<td>5400</td>
<td>Yellow Green</td>
</tr>
<tr>
<td>5600</td>
<td>GreenYellow</td>
</tr>
<tr>
<td>5700</td>
<td>Yellow -&quot;Yellowish&quot;(at times appears to be Lt gray or metallic)</td>
</tr>
<tr>
<td>5800</td>
<td>Light Orange or Yellow - Pink</td>
</tr>
<tr>
<td>6000</td>
<td>Carnation Pink</td>
</tr>
<tr>
<td>6300</td>
<td>Violet Red</td>
</tr>
<tr>
<td>6800</td>
<td>&quot;Bluish&quot;(appears violet red, Blue Green, looks)</td>
</tr>
<tr>
<td>7200</td>
<td>Blue Green - Green</td>
</tr>
<tr>
<td>7700</td>
<td>&quot;Yellowish&quot;</td>
</tr>
<tr>
<td>8000</td>
<td>Orange</td>
</tr>
<tr>
<td>8200</td>
<td>Salmon</td>
</tr>
<tr>
<td>8500</td>
<td>Dull, Light Red Violet</td>
</tr>
<tr>
<td>8600</td>
<td>Violet</td>
</tr>
<tr>
<td>8700</td>
<td>Blue Violet</td>
</tr>
<tr>
<td>8900</td>
<td>Blue</td>
</tr>
<tr>
<td>9200</td>
<td>Blue Green</td>
</tr>
<tr>
<td>9500</td>
<td>Dull Yellow Green</td>
</tr>
<tr>
<td>9700</td>
<td>Yellow - &quot;Yellowish&quot;</td>
</tr>
<tr>
<td>9900</td>
<td>Orange</td>
</tr>
<tr>
<td>10000</td>
<td>Carnation Pink</td>
</tr>
</tbody>
</table>

Nitride Thickness = (Oxide Thickness)(Oxide Index/Nitride Index)
Eg. Yellow Nitride Thickness = (2000)(1.46/2.00) = 1460
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.
Approximately 1 gm of Parylene C gives ~3000Å 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](http://www.scscookson.com/parylene/properties.cfm)
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 pealing.
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
