Goal: Understand the impact of MBE process variables on the stoichiometric performance indicators of MgO films through DOE based analysis on historical experimental data

Data, Tools and Techniques

- XPS analysis of historic data (180 samples)
- Neural Networks to model MBE process
- Full factorial design for experiments (DOE)
Taking microelectronics beyond silicon:

Integrating functional oxides with wide bandgap semiconductors to meet high-power, high-temperature, high-frequency applications

Engineering Structure and Chemistry Atom by Atom

• Clearer Cell Phone Receptions with Fewer Towers
• Computer Displays that Roll-up like a Newspaper
• Hand-held integrated functionality
• Self-Driving Cars
• Quantitative Biosensors
• Smart Energy

Northeastern University
The Chemistry and Structure of MgO and its Role as an Interface Layer

BTO(111)/MgO(111)/6H-SiC(0001)

BTO(111)
O to O 2.83 Å
-8.83% / -5.3%

MgO(111)
O to O 2.98 Å

SiC(0001)
Si to Si 3.08 Å

Blue = O (from BTO)
Red = O (from MgO)
Green = Si (from SiC)
MgO Growth by MBE (Molecular Beam Epitaxy)

Ultra-High Vacuum (UHV)

- $10^{-9}$ to $10^{-10}$ Torr
- km mean free path
- 45 minutes for monolayer coverage
- literally atomic/ particle level control

UHV and MBE offer atomic level control over the growth conditions and processing parameters necessary for engineering an effecting interface.
Equipment, Reaction and Controls

\[ \text{Mg (mixed flux)} + \text{O (plasma)} \xrightarrow{\Delta \ UHV} \text{Mg-O} \]

- Low-temperature effusion cells (Mg)
- OAR rf-plasma source (O & O\(_2\))
- Substrate heater (up to 900 °C)

UHV Analysis – Surface Structure
- Reflection High-Energy Electron Diffraction (RHEED)

UHV Analysis – Surface Chemistry
- X-ray Photoelectron Spectroscopy (XPS)
- Auger Electron Spectroscopy (AES)
Equipment, Reaction and Controls

What is necessary:
- Low plasma power
- Low temperature
- Mg-adsorption controlled growth
- Slow rates

UHV Analysis – Surface Chemistry
- X-ray Photoelectron Spectroscopy (XPS)
- Auger Electron Spectroscopy (AES)
Research Approach

Vary Process Parameters

UHV Processing (Loop 1)

Growth Mechanisms

Film Chemistry & Structure

Engineered Chemistry Structure

Performance Properties of Film

UHV ?? Processing (Loop 2)

Vary Process Parameters

Tuned Performance
Based on experimental historic data develop a process model to map, analyze and control the process.
Details of the Experiment

Degreasing and hydrogen cleaning of 6H-SiC substrate.

Sample loaded into UHV chamber.

In-situ Chemical and structural characterization by XPS and RHEED.

Sample transported to Growth chamber.

Mg effusion cell and substrate heating and oxygen plasma generation with controlled chamber pressure.

In-situ Chemical and structural characterization by XPS and RHEED.

MgO growth

Maintaining high vacuum.

Maintaining high vacuum.

UHV and MBE offer atomic level control over the growth conditions and processing parameters necessary for engineering an affecting interface.
X-Ray Photoelectron Spectroscopy (XPS)

- Elemental Identification
- Chemical State Identification
- Elemental Quantification

- Understand the chemistry of starting and final surfaces.
- Engineer processes to grow single crystal, epitaxial, stoichiometric oxide thin films with desired functional properties.

Schematic diagram of the principle of XPS
XPS Spectra of SiC after Hydrogen Cleaning

Composition:
Si : 49.0%
C : 43.2%
O : 7.8%
XPS Spectra of ~2 nm MgO Film on SiC Substrate

Composition:
Si: 18.3%
C: 29.7%
O: 29.9%
Mg: 22.1%

Mg-O
Mg-OH
O-Mg
OH-Mg
Si-C
Si-Ox
Input-Process-Output Diagram

- Mg + O\textsubscript{(plasma)} $\xrightarrow{\Delta}$ MgO
- Plasma Pressure
- Plasma Power
- Mg Source Temperature
- Substrate Temperature
- Growth Time
- Oxygen as percentage composition after cleaning.
- Plasma Intensity
- O-Mg / O
- OH-Mg / O
- (O-Mg + OH-Mg )/O

Magnesium
Oxygen Plasma
SiC Substrate

UHV and MBE offer atomic level control over the growth conditions and processing parameters necessary for engineering an effective interface.
Full Factorial Design of Experimentation and DOE Procedure

- Complexity of process
- Number of variables
- Number of data points

Three factor three level design space (3k).

http://www.mathworks.com/access/helpdesk/help/toolbox/stats
MgO Growth Data and its Limitations

Inputs

- Growth time (C)
- Substrate temperature (C)
- Mg source temperature (C)
- Plasma pressure (C)
- Plasma Power (C)
- Plasma Intensity (UC)
- Oxygen as % of cleaned surface (UC)

Outputs

- O-Mg : O
- OH-Mg : O
- (O-Mg + OH-Mg) : O

Issues

- Experiments were not designed for DOE
- Large number of variables
- We had to rely on oxygen peak for enough data points

C = controllable process variable
UC = uncontrollable process variable
The Model

Historical Data
(180 non statistically designed experiment)

Training Neural Network
(180 Input patterns with 3 different outputs)

Elimination of Outliers
(Same data presented to Network and 5% input patterns towards the right tail of normally distributed corresponding error plot removed as outliers)

Training Neural Network
(Balance of remaining patterns again presented to neural network with same configuration)

Generate Full Factorial DOE Runs for 10 Replications
(Given variables, their min max, mid point values and designing 3 level experiments; input patterns were generated and 10 replication of each pattern were created to add some Gaussian noise to the data)

Estimate Process Response to DOE Runs Using NN
(DOE based input patterns to the trained network and recorded the predicted outputs)

DOE Analysis
(Analysis of data set against their respective predicted outputs from the neural network trained on original data)
Utilization and Response of Model to the Data

180 sample Data → NN learns the data → Test NN with Data → Eliminate Top 5% outliers

Create full-factorial DOE runs for 10 replications

NN predicts the response for the DOE runs → Perform DOE Analysis. → Eliminate sample data based on the DOE analysis, 155 to 104.

Test NN with Data → NN learns the remaining 155 Data

Testing with 8 input variables → Testing with 7 input variables → Testing with 6 input variables

Critical variables finalized and establishment of empirical relation with the outputs.
Elimination of 5% Outliers

Before 180 Samples

After 155 samples

Residuals/Outliers
Elimination of Sample Data

Identify the range for individual variables based on their density.
Modification of DOE

- Reducing Number of Samples from 155 to 104 by eliminating experiments that are radically different from the rest

- Reducing the number of variables from 8 to 7 by eliminating the redundant variables

- Adjust the min and max settings of each variables by observing the density of data distribution
Marginal Mean Plots of 104 Samples (7 Variables)

- Growth time (C)
- Substrate temperature (C)
- Mg source temperature (C)
- Plasma pressure (C)
- Plasma Power (C)
- Plasma Intensity (UC)
- Oxygen as % of cleaned surface (UC)
Response Surface Plots of O-Mg and OH-Mg
(Substrate Temperature and % Oxygen on clean surface)
Response Surface Plots of O-Mg and OH-Mg (Zoom In)

Growth Time and Plasma Power
Results and Discussion

• Higher temperature means more molecular movement. Makes surface smoother and ensures 2D growth. That is why we see lesser OH-Mg.
• Increasing % oxygen O-Si results in less O-Mg and more OH-Mg, which refers to more 3D growth.
• Helps assert that OH-Mg comes from substrate. As the film grows thicker we cannot see that.
• Higher plasma power results in more O-Mg.
• The model can help save cost of experimentation and achieve process control.
• Model can analyze more than one key performance indicators.
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