DOE Based Analysis of Magnesium Oxide Film Grown by Molecular Beam Epitaxy

(surface composition indicators)

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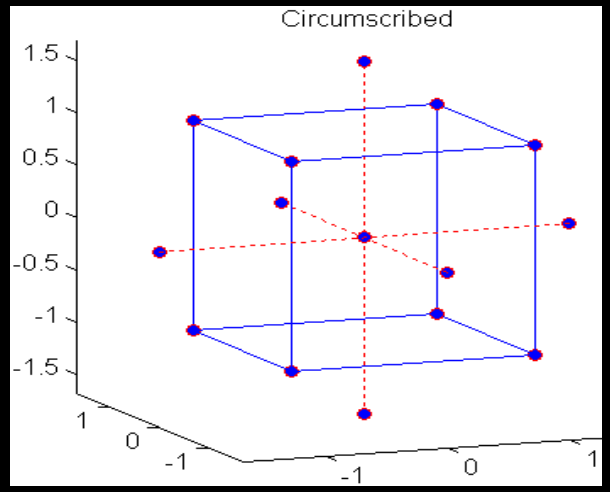
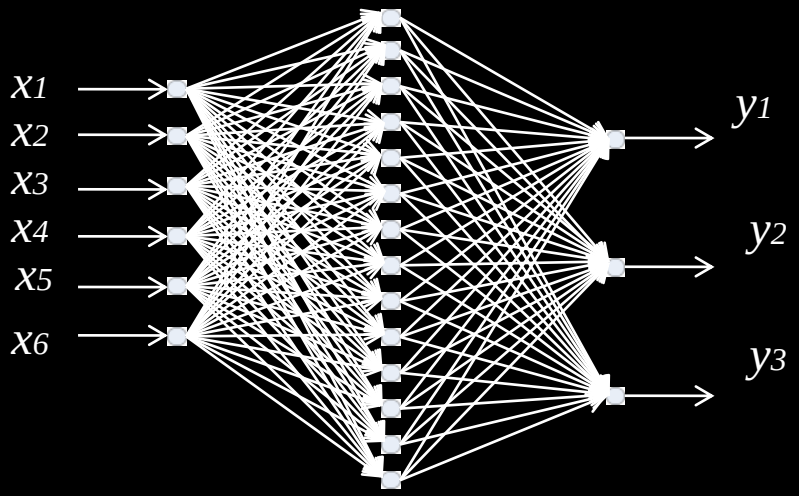
Goal: Understand the impact of MBE process variables on the stoichiometric performance indicators of MgO films through DOE based analysis on historical experimental data

Data, Tools and Techniques

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





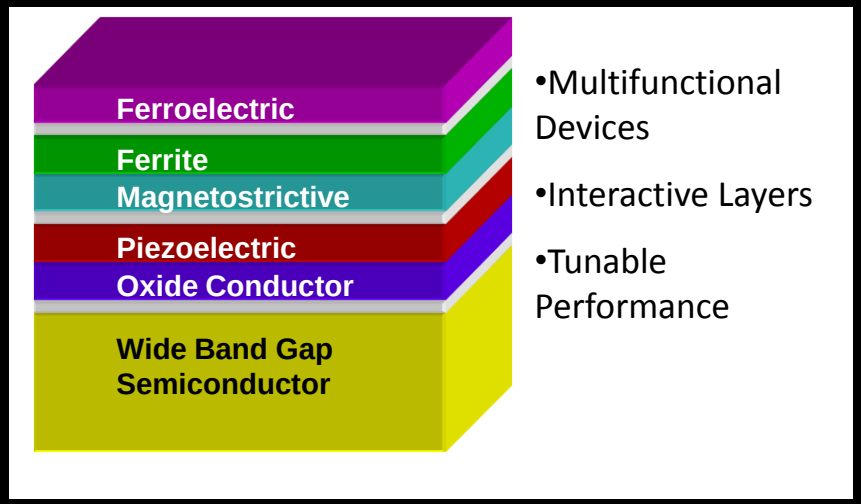
Semiconductors, Functional Oxide Hetro-Structures, and MgO on 6H-SiC

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



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

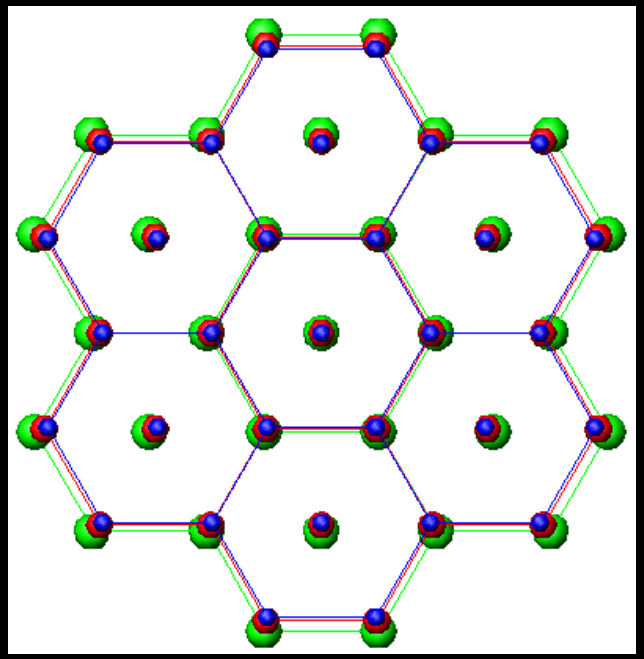
BTO(111)/MgO(111)/6*H*-SiC(0001)

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)

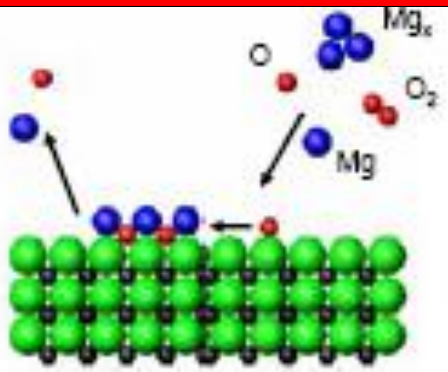
Red = O (from MgO)

Green = Si (from SiC)

MgO Growth by MBE (Molecular Beam Epitaxy)

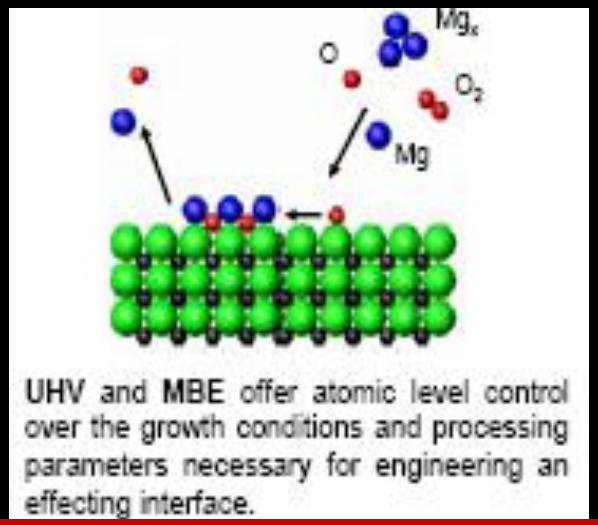
Ultra-High Vacuum (UHV)

- 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



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

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

- Low-temperature effusion cells (Mg)
- OAR rf-plasma source (O & O_2)
- Substrate heater (up to 900 ^{o}C)

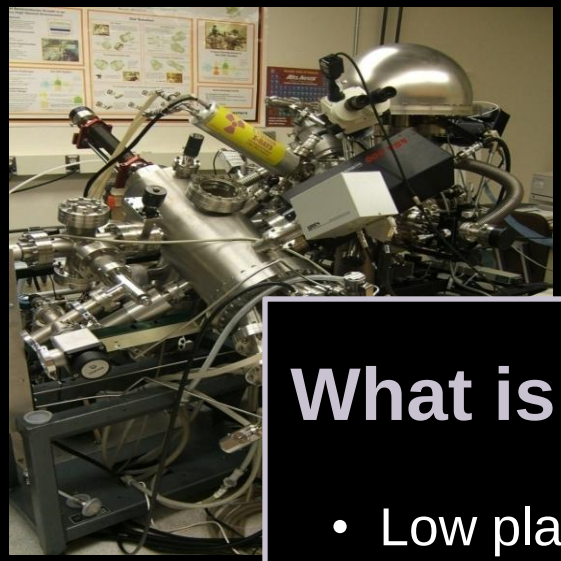
UHV Analysis – Surface Structure

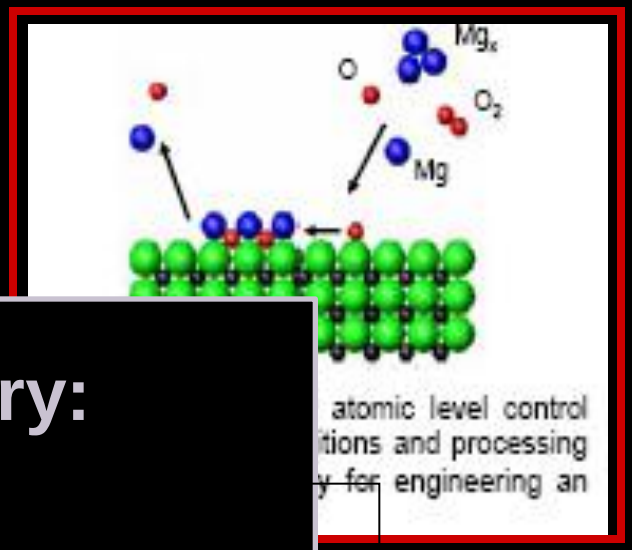
- **R**eflection **H**igh-**E**nergy **E**lectron **D**iffraction (RHEED)

UHV Analysis – Surface Chemistry

- **X**-ray **P**hotoelectron **S**pectroscopy (XPS)
- **A**uger **E**lectron **S**pectroscopy (AES)

Equipment, Reaction and Controls





What is necessary:

- Low plasma power
- Low temperature
- Mg-adsorption controlled growth
- Slow rates

Mg *(mixed*

- Low-temperatur

- OAR rf-plasma source (O & O_2)

- Substrate heater (up to 900 ^{o}C)

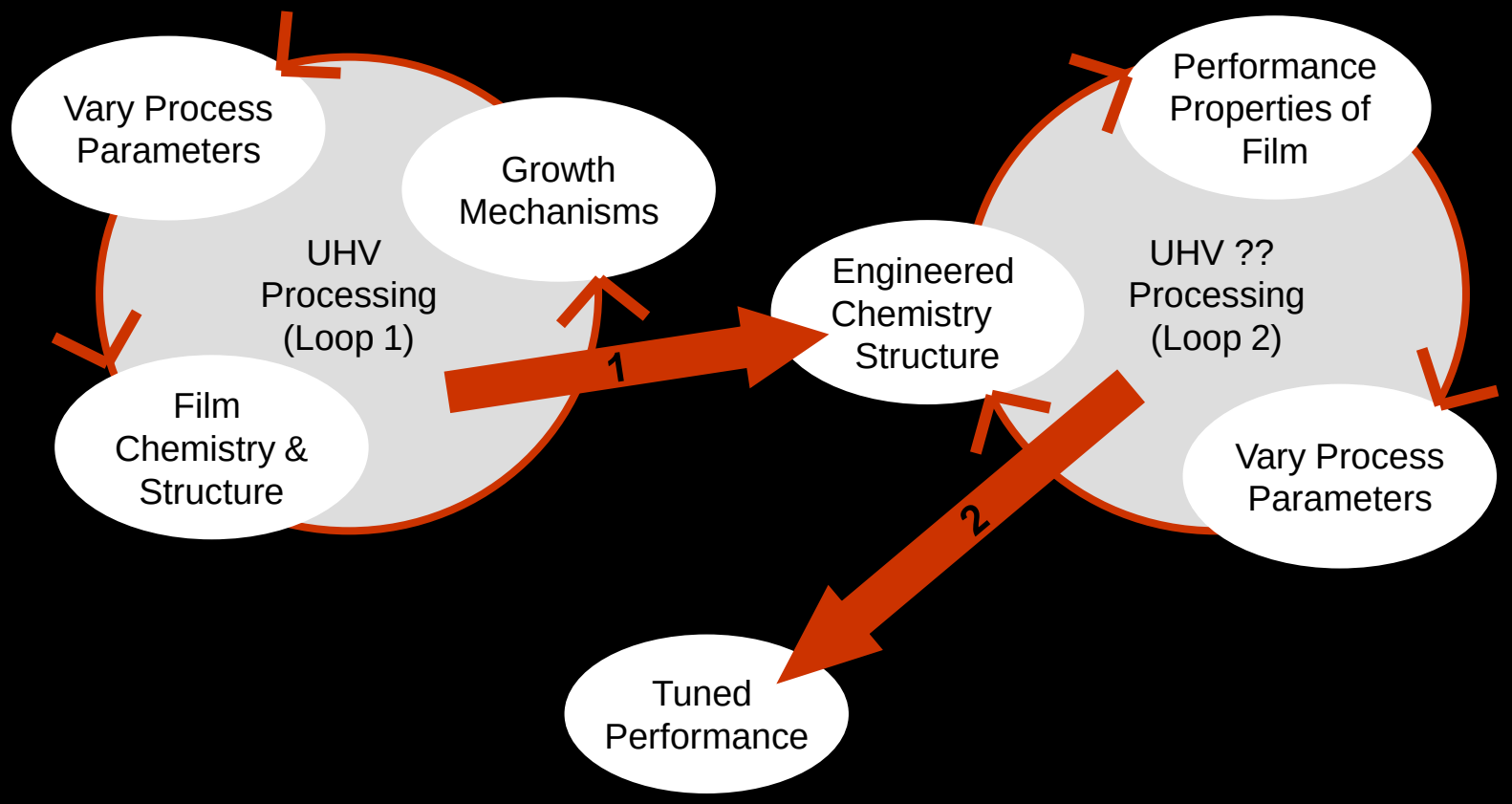
face Structure

-Energy Electron
ED)

UHV Analysis – Surface Chemistry

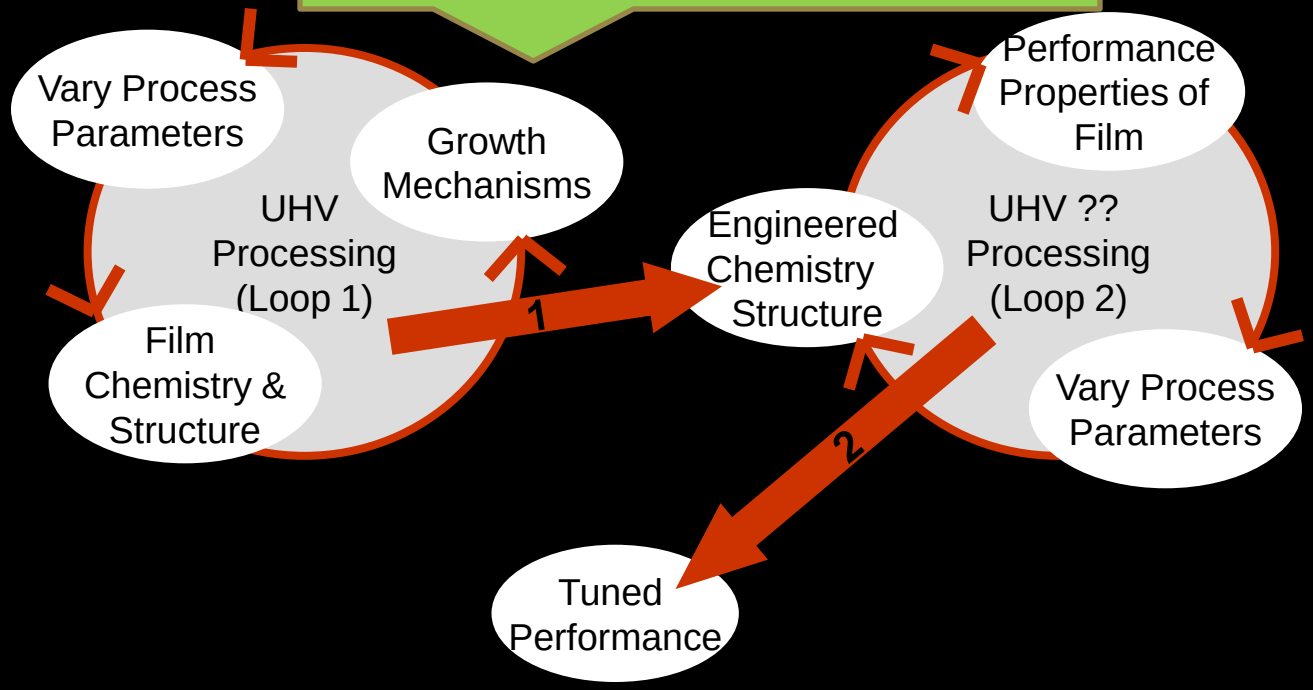
- **X**-ray **P**hotoelectron **S**pectroscopy (XPS)
- **A**uger **E**lectron **S**pectroscopy (AES)

Research Approach



Analysis Based on Historic Data

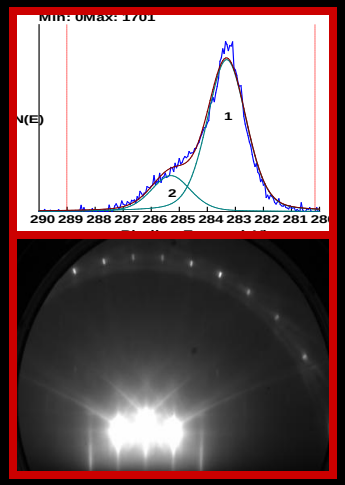
Based on experimental historic data develop a process model to map, analyze and control the process



Details of the Experiment

Degreasing and hydrogen cleaning of 6H-SiC substrate.

Sample loaded into UHV chamber.

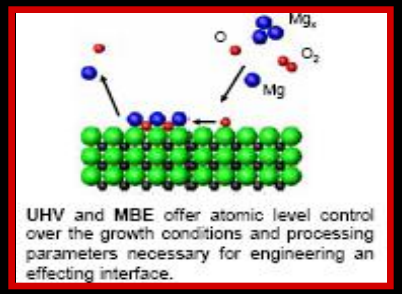


In-situ Chemical and structural characterization by XPS and RHEED.

Sample transported to Growth chamber.

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

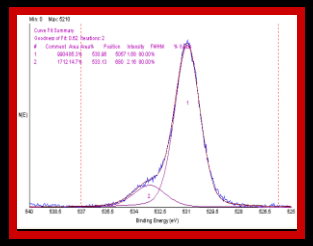
MgO growth

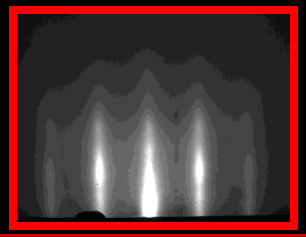


Maintaining high vacuum.

Maintaining high vacuum.

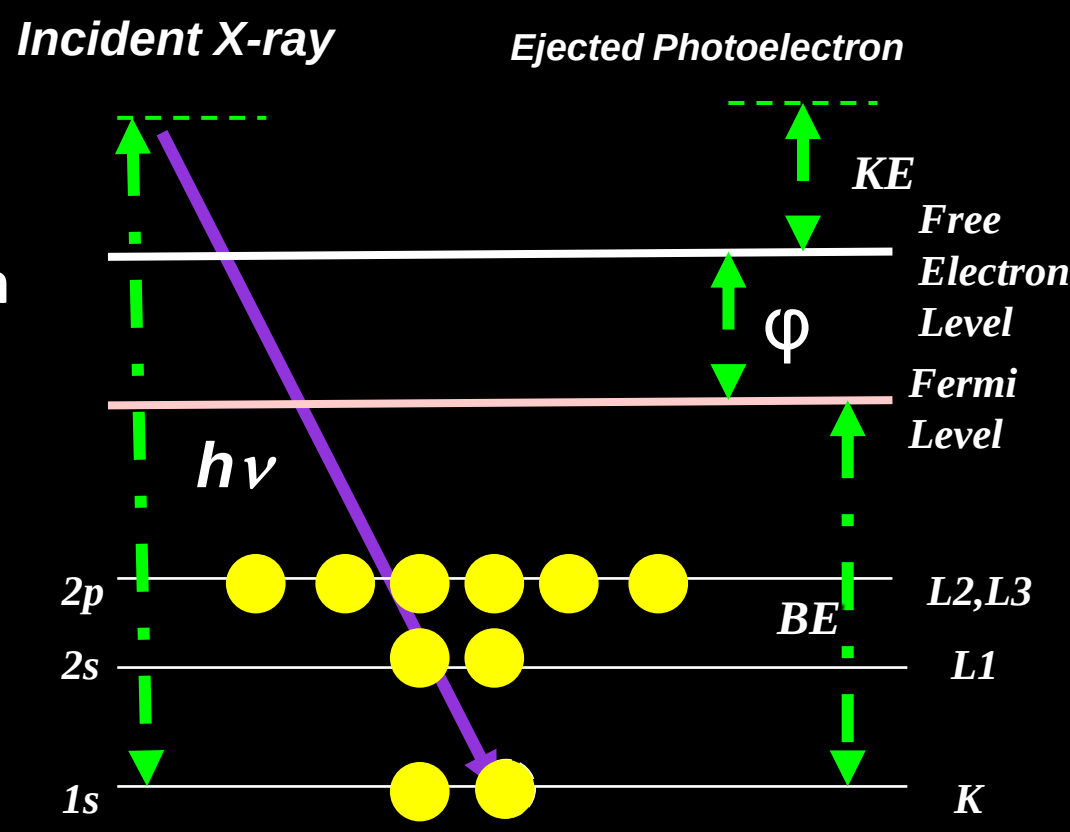
In-situ Chemical and structural characterization by XPS and RHEED.





X-Ray Photoelectron Spectroscopy (XPS)

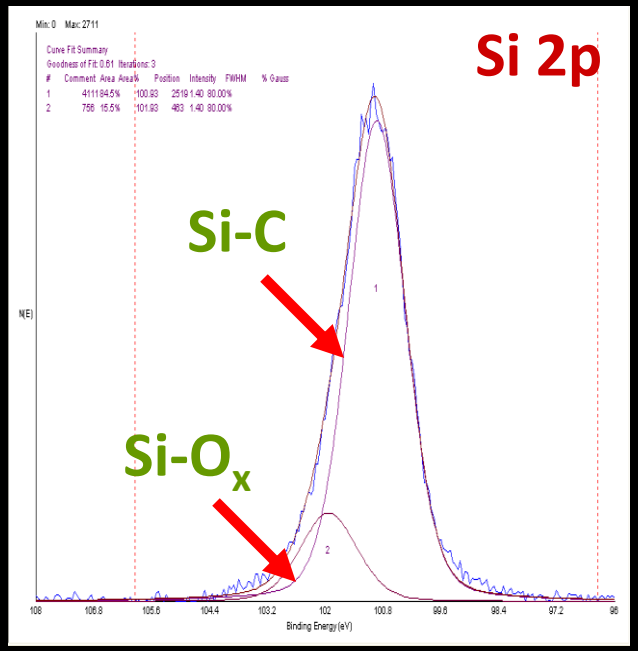
- ✓ Elemental Identification
- ✓ Chemical State Identification
- ✓ Elemental Quantification

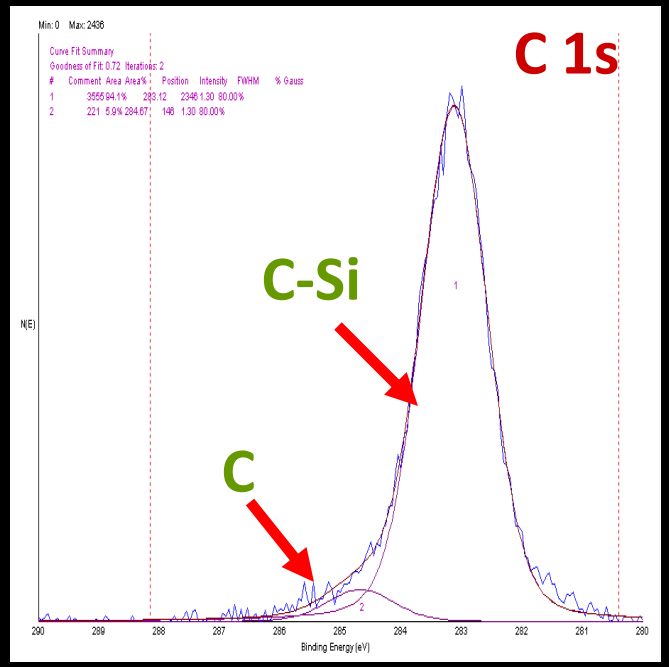


Schematic diagram of the principle of XPS

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

XPS Spectra of SiC after Hydrogen Cleaning



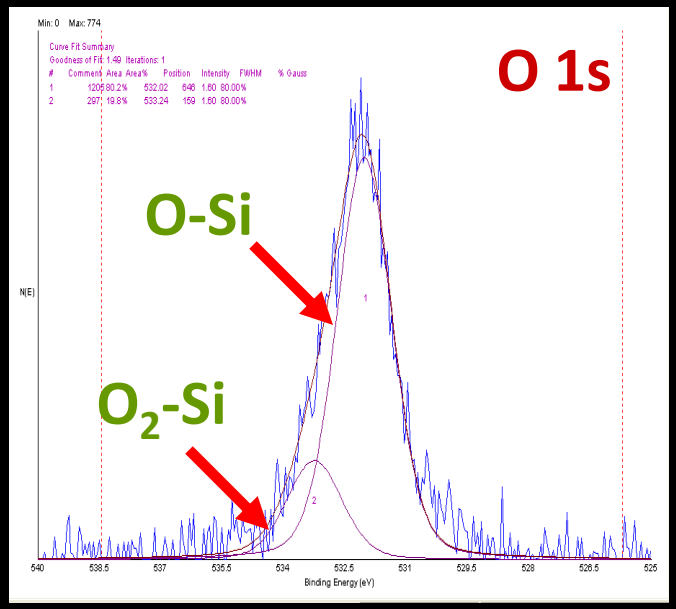


Composition:

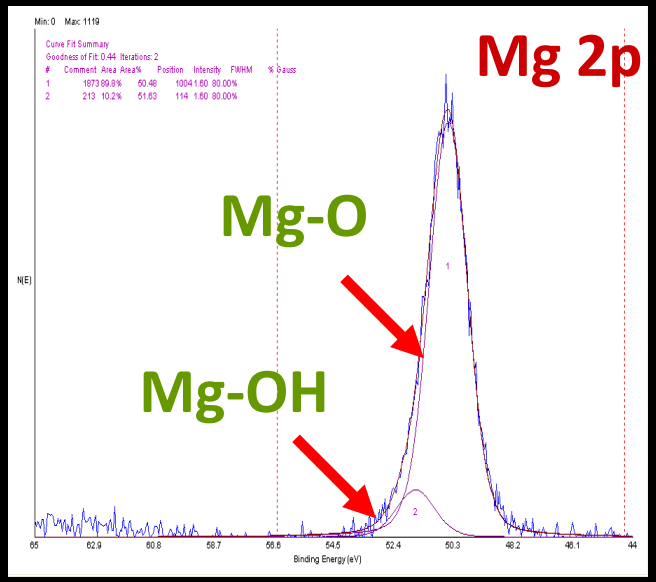
Si : 49.0%

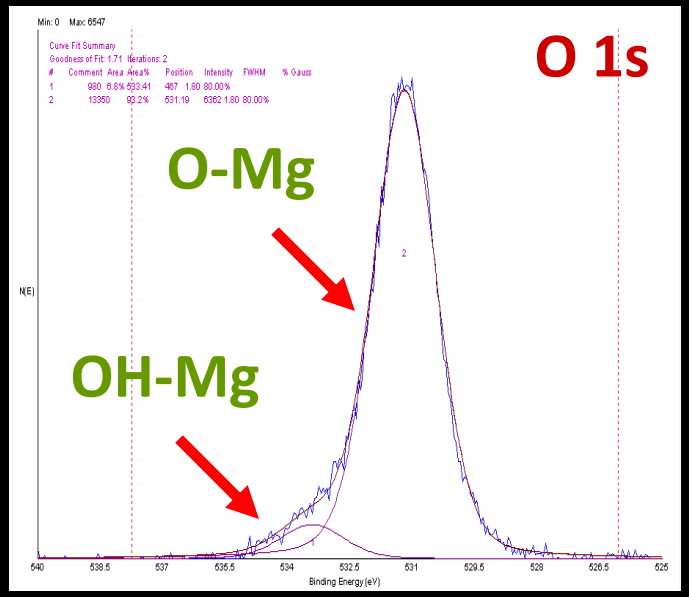
C : 43.2%

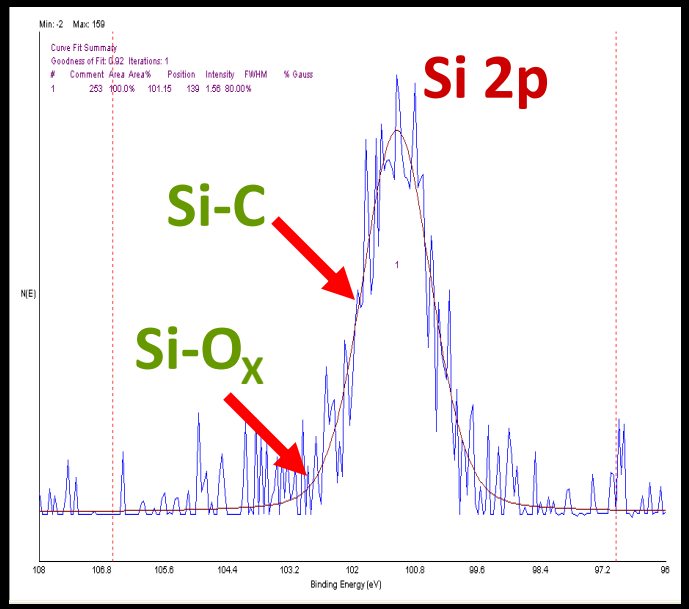
O : 7.8%



XPS Spectra of ~2 nm MgO Film on SiC Substrate







Composition:

Si: 18.3%

C: 29.7%

O: 29.9%

Mg: 22.1%

Input-Process-Output Diagram

Plasma Power

Plasma Pressure

Growth Time

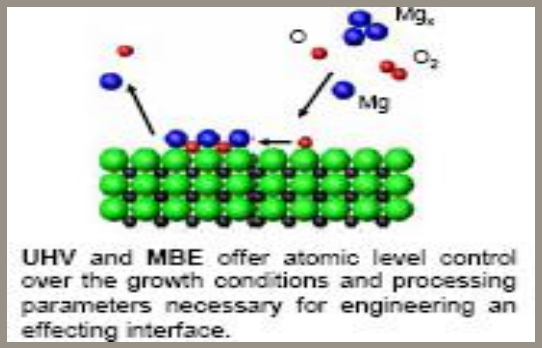
Mg Source Temperature

Substrate Temperature

Magnesium

Oxygen Plasma

SiC Substrate



$$Mg + O_{(plasma)} \xrightarrow{\Delta} MgO$$

O-Mg / O

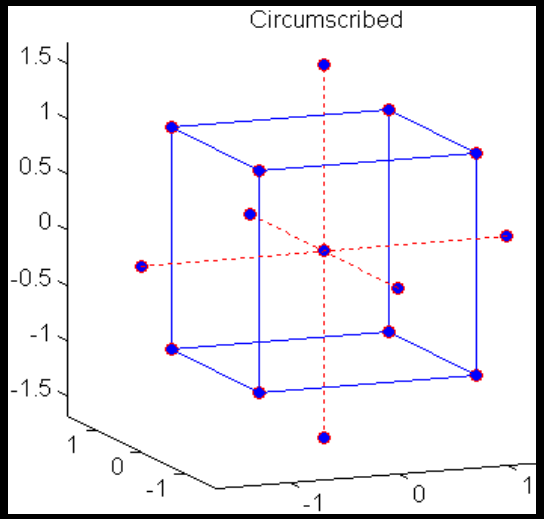
OH-Mg / O

(O-Mg + OH-Mg)/O

Oxygen as percentage composition after cleaning.

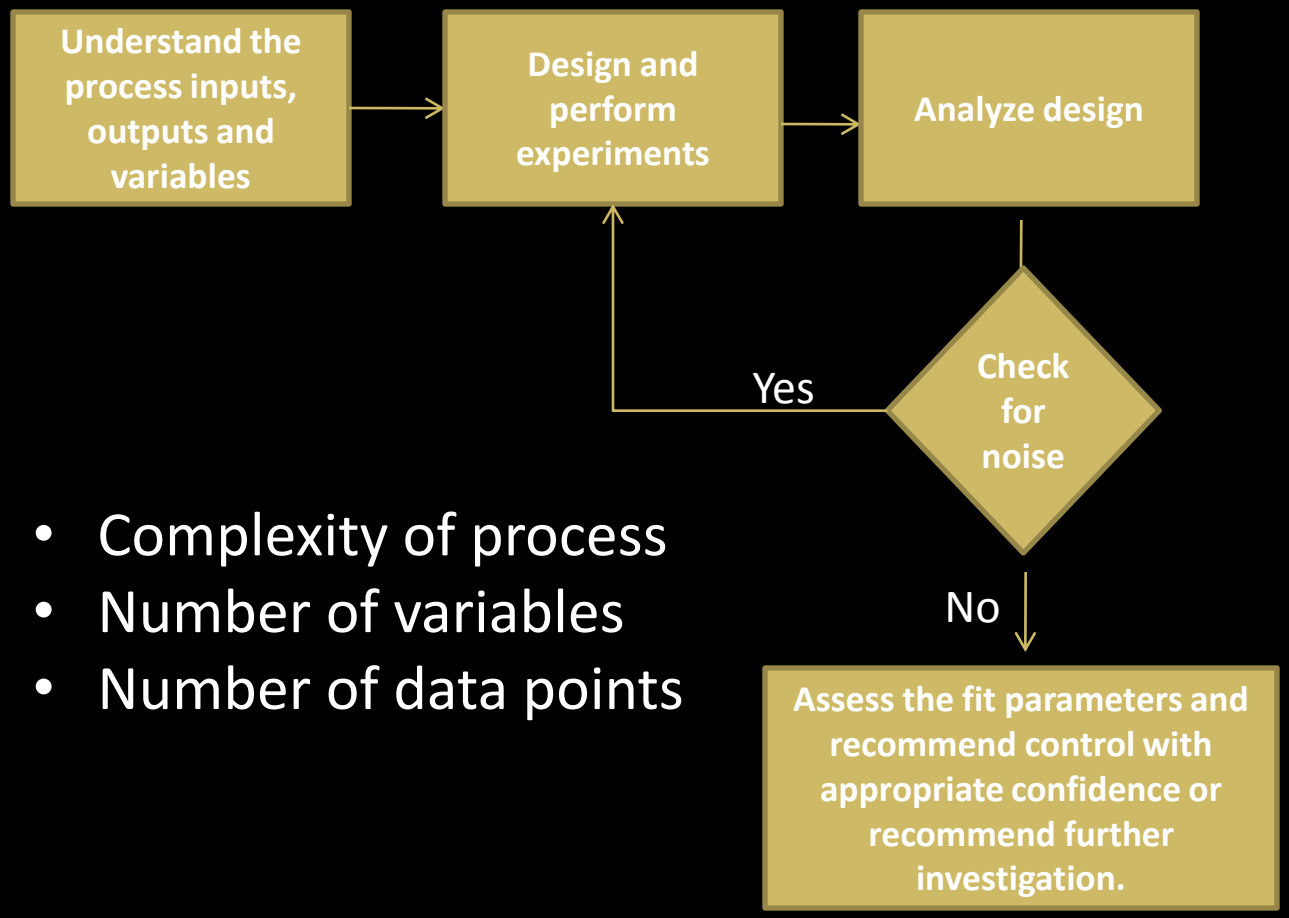
Plasma Intensity

Full Factorial Design of Experimentation and DOE Procedure



Three factor three level design space (3k).

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



http://www.mathworks.com/access/helpdesk/help/toolbox/stats

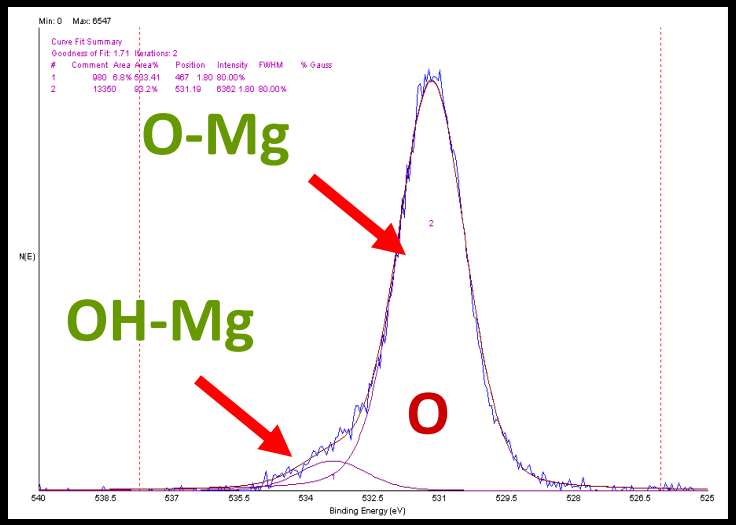
MgO Growth Data and its Limitations

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)

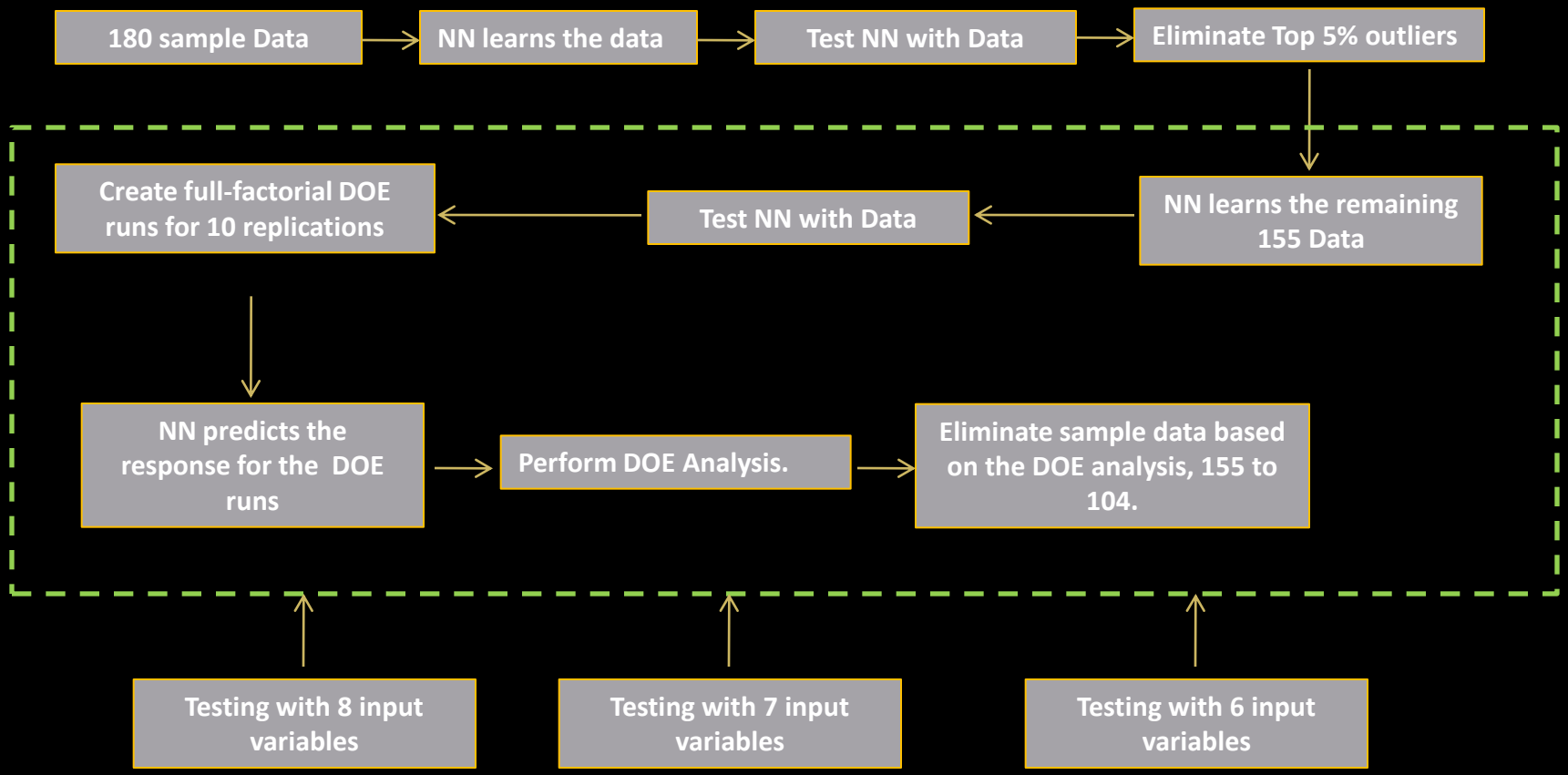
Training Neural Network
(Balance of remaining patterns again presented to neural network with same configuration)

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)

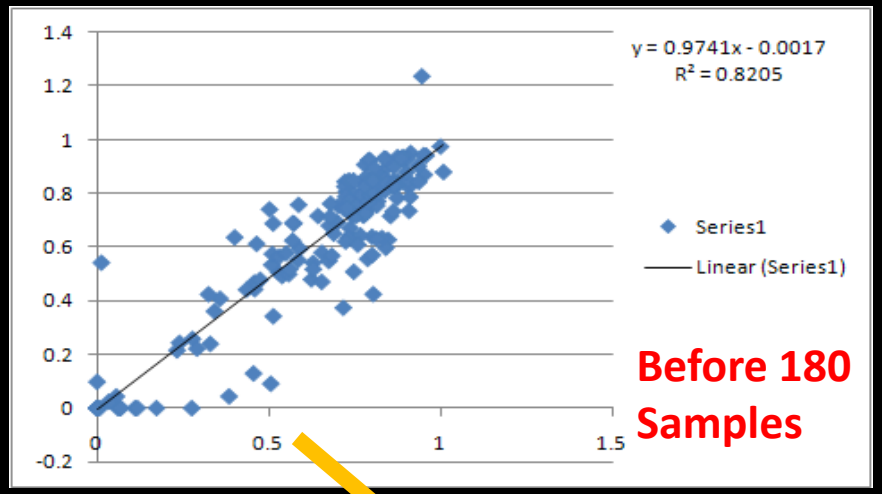
DOE Analysis
(Analysis of data set against their respective predicted outputs from the neural network trained on original data)

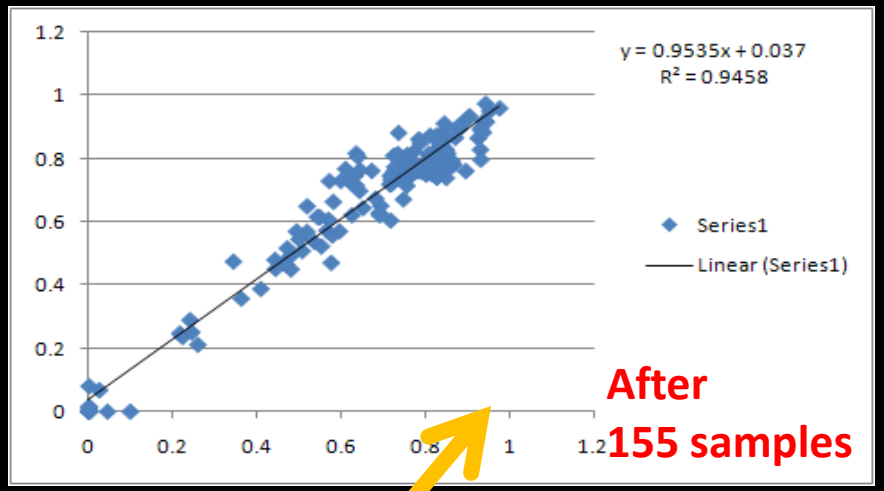
Utilization and Response of Model to the Data

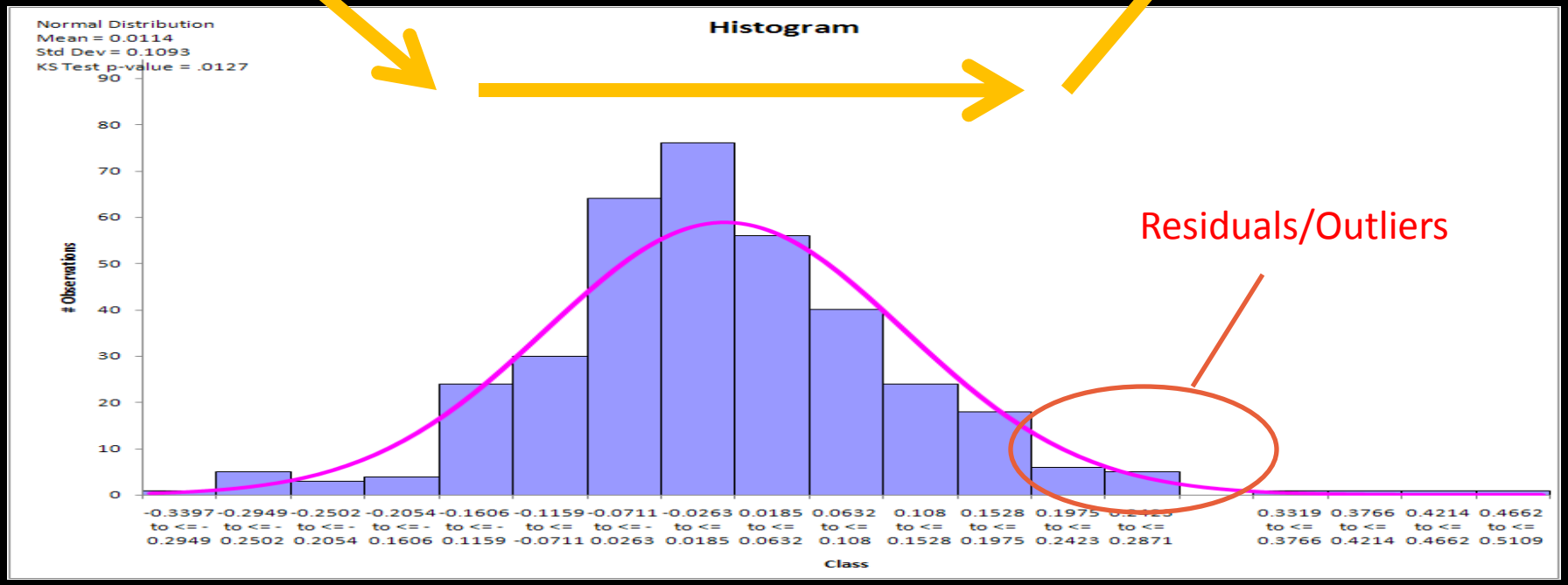


Critical variables finalized and establishment of empirical relation with the outputs.

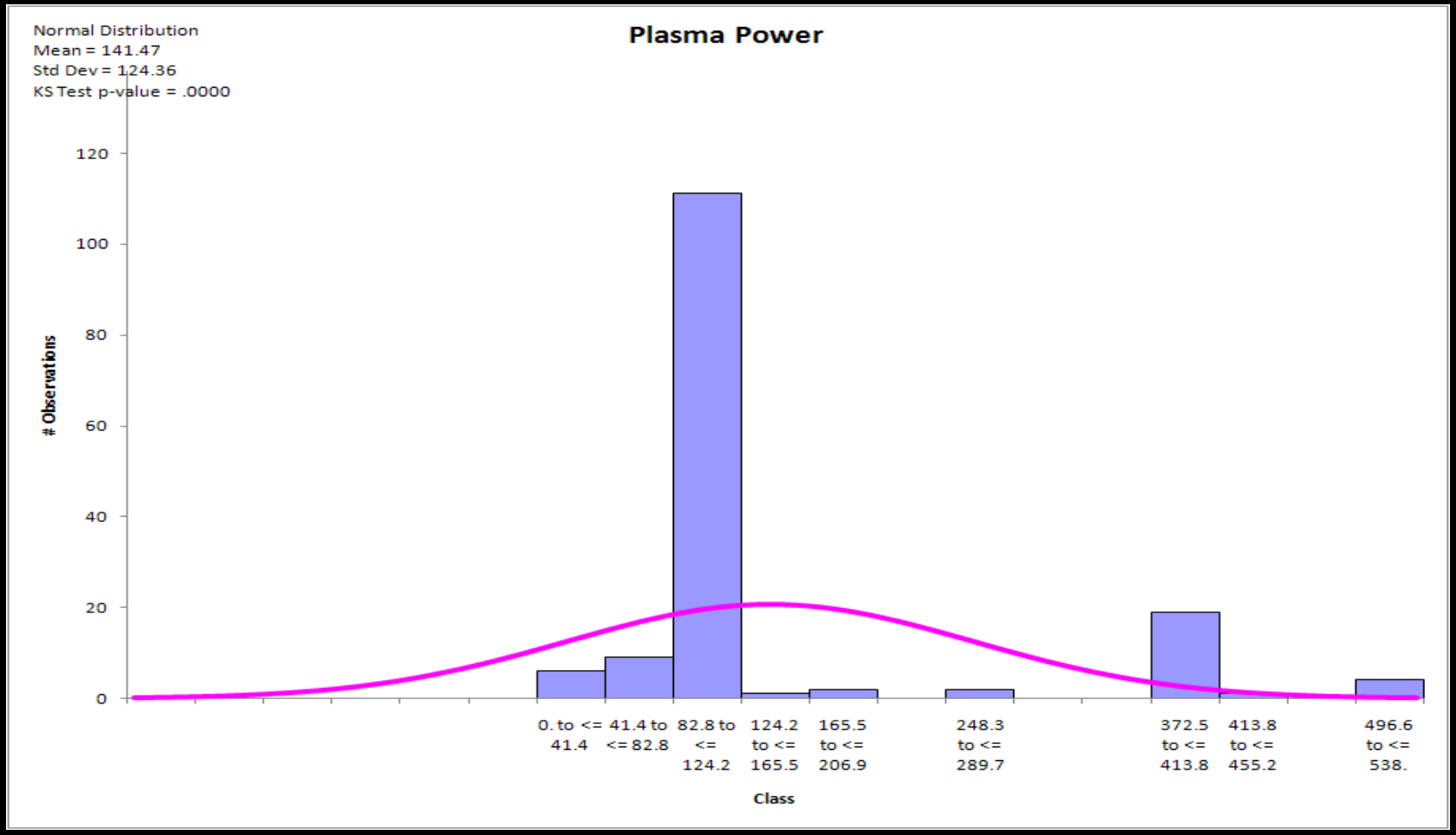
Elimination of 5% Outliers







Elimination of Sample Data



Identify the range for individual variables based on their density

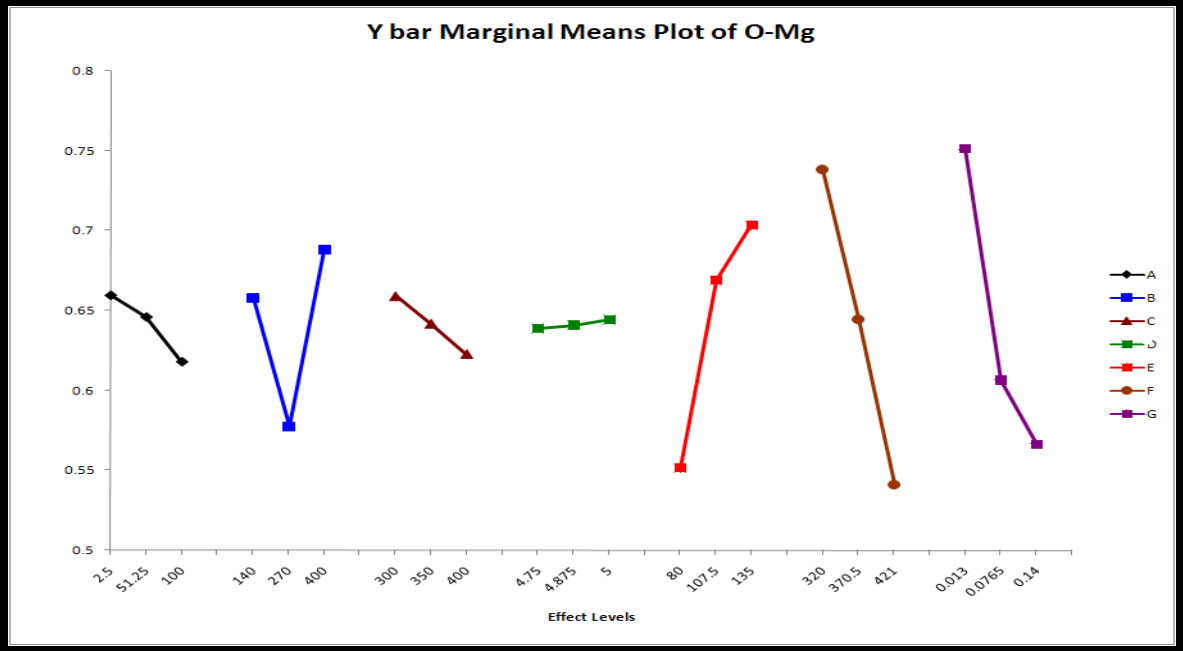
Modification of DOE

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

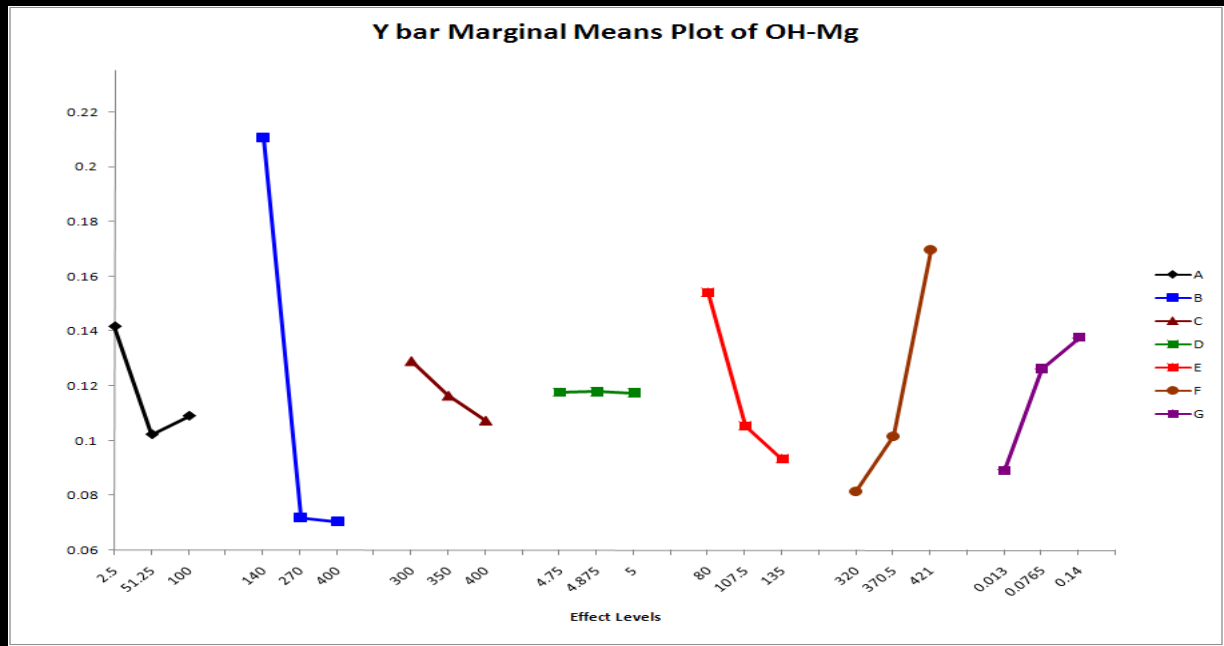
Mg source temperature (C)

Plasma pressure (C)

Plasma Power (C)

Plasma Intensity (UC)

Oxygen as % of cleaned surface (UC



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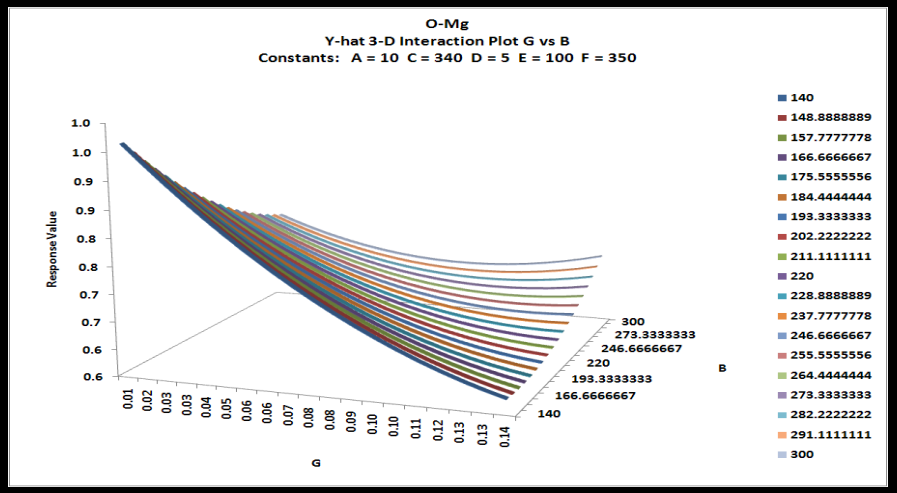
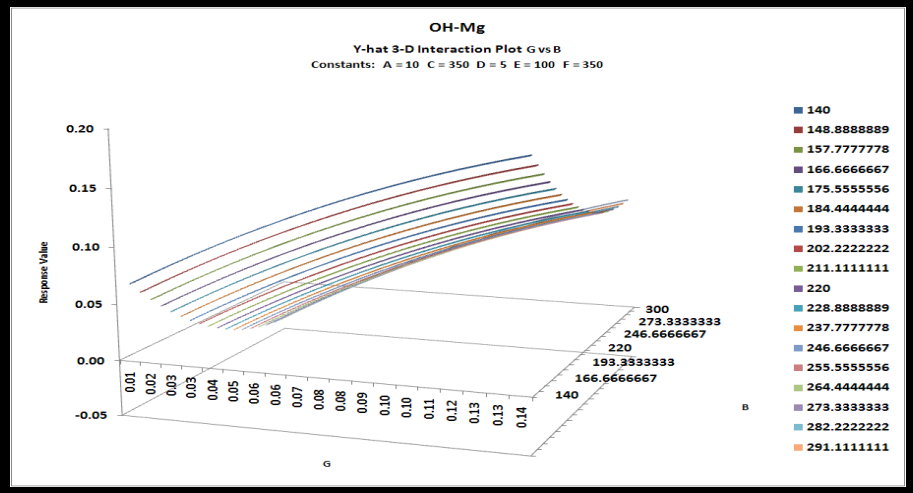
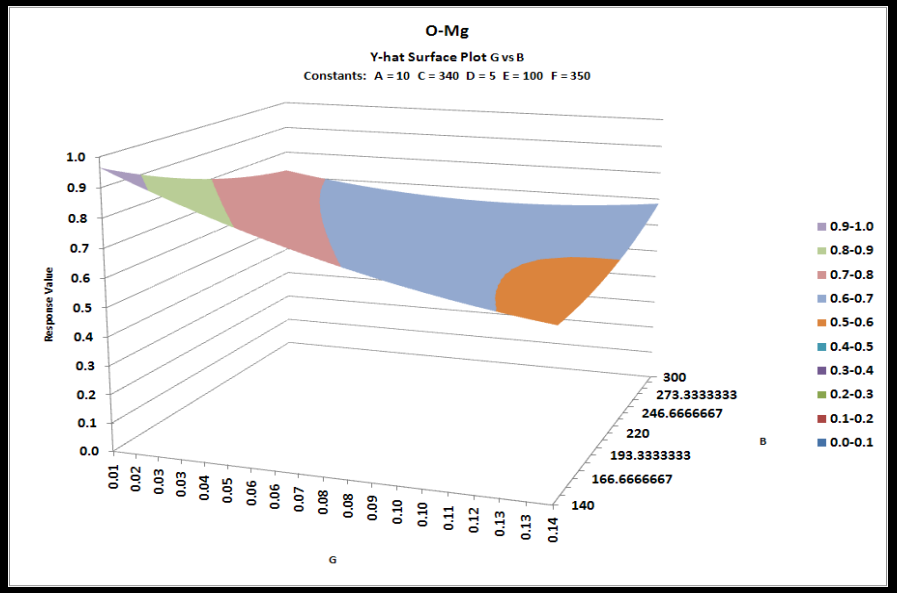
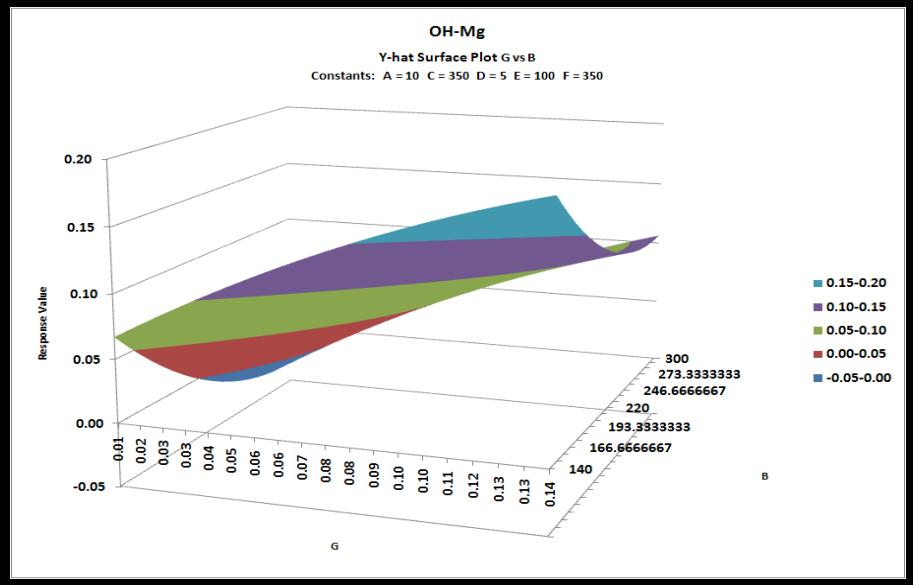
Response Surface Plots of O-Mg and OH-Mg

(Substrate Temperature and % Oxygen on clean surface)



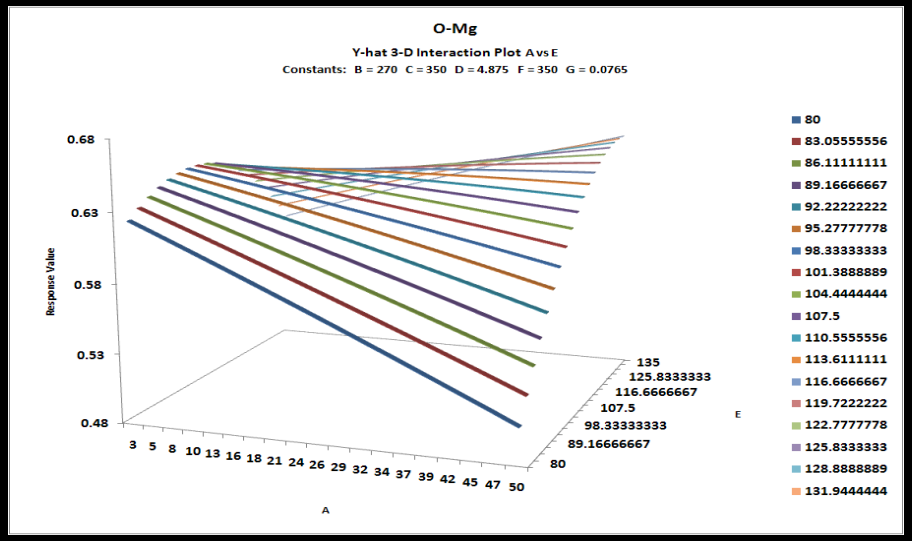


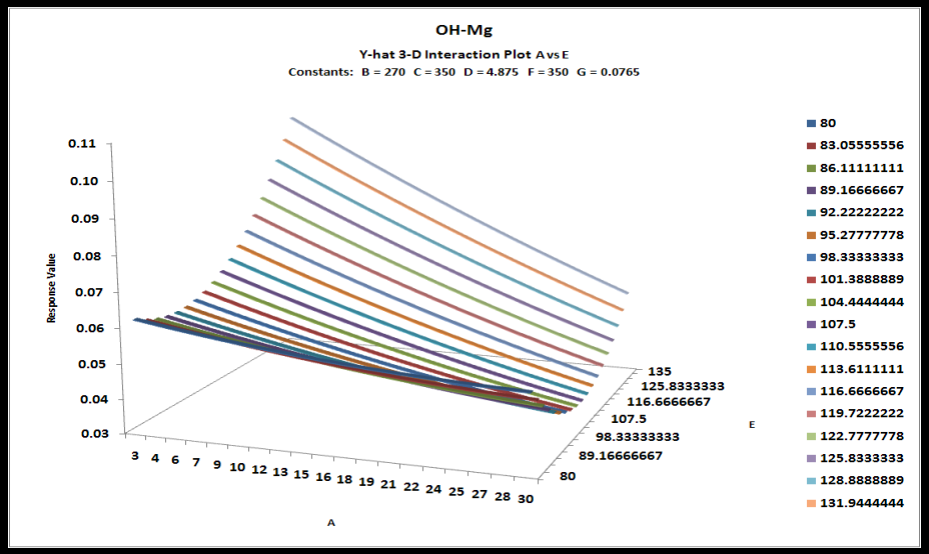


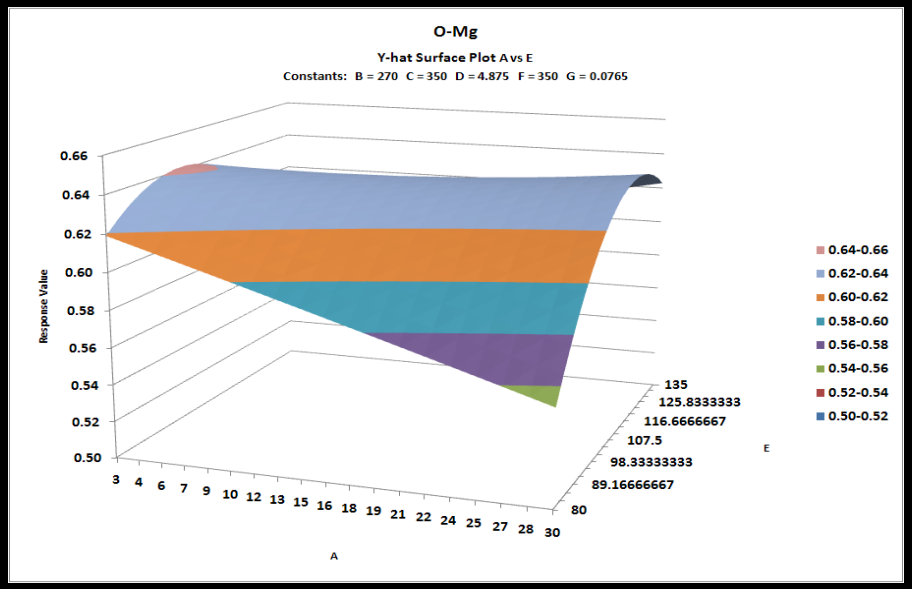


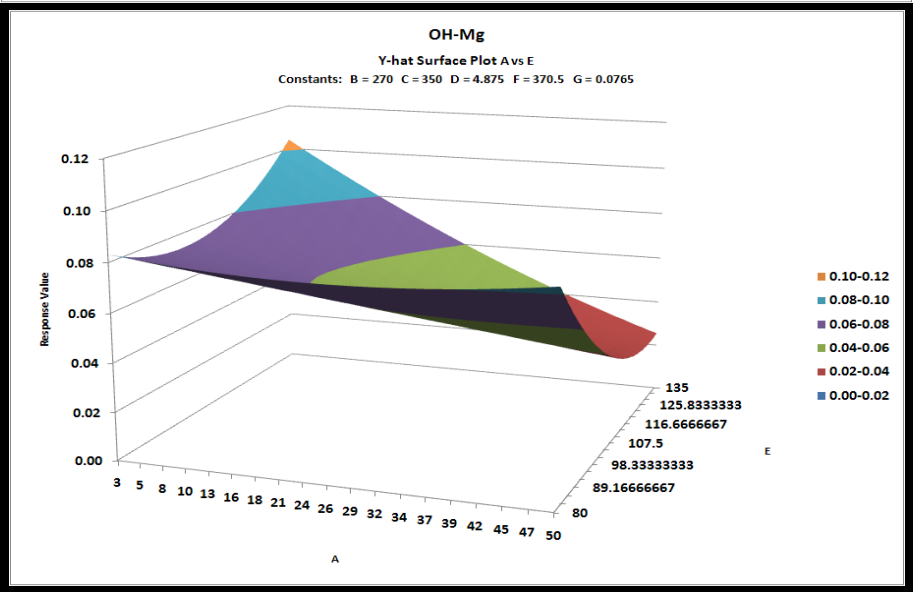
Response Surface Plots of O-Mg and OH-Mg (Zoom In)

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.

Acknowledgments

Interface Engineering Laboratory

- **Dr. Katherine S. Ziemer**
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- **Bing Sun**
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