Ellipsometry and XPS for Thin Film Aluminum Analysis

18th Annual Mirror Technology SBIR/STTR Workshop

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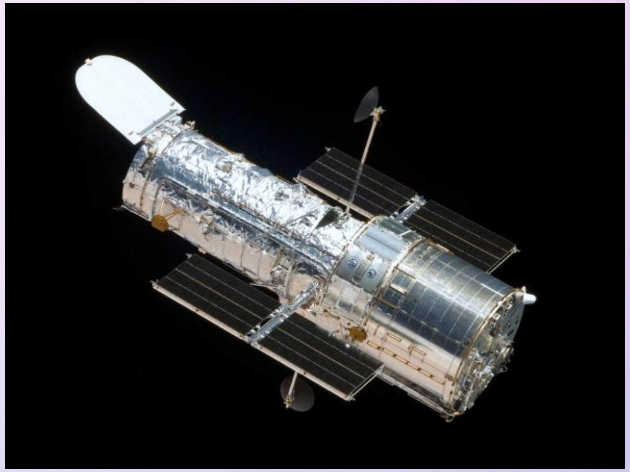
Aluminum as a Reflector

- NASA (Goddard Space Flight Center)
  - Currently developing LUVIOR (Large, UV-optical-IR)
- LUVOIR
  - NASA observatory of the 2020's or 30's
  - Largest mirrors ever put in space
  - Being designed to operate in
    - Ultraviolet regions
    - Visible Regions
    - Infrared Regions
  - Reflective coating will be Aluminum



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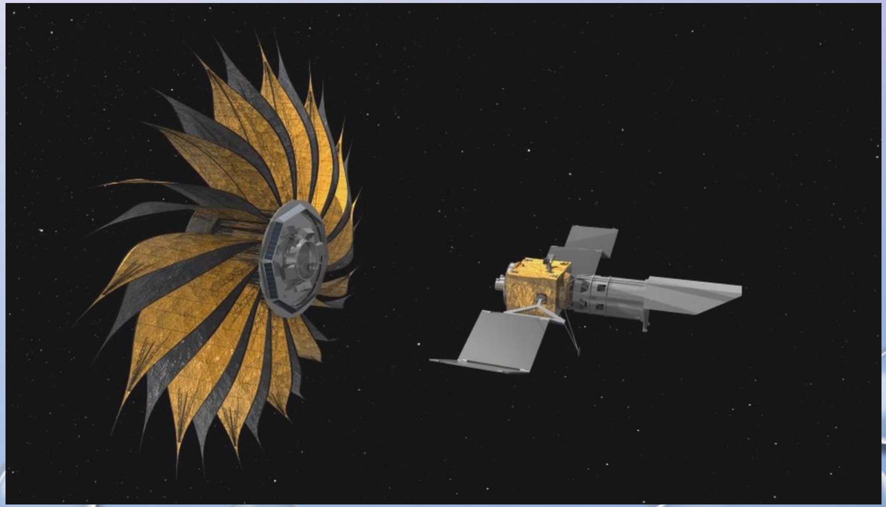

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Aluminum as a Reflector

- **Pros of Aluminum**
  - **Inexpensive**
  - **Easy to work with**
    - **Depositions are trivial**
    - **Deposition is reproducible**
  - **Superior reflective material**
    - **Widest bandwidth of reflectivity of all materials**
- **Cons**
  - **Aluminum begins oxidizing instantly**
  - **Oxidation causes optical losses and attenuation-UV**

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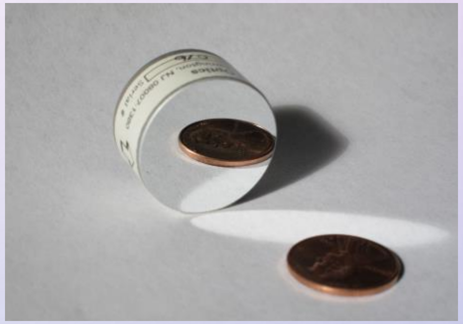

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Protection of Aluminum

- Barrier layers
  - Fluoride thin films
  - Transparent
  - MgF_2 is generally used
    - Wide bandgap
    - Minimal optical interference
    - 25 nm is used
  - Other fluorides
    - LiF
    - AlF_3
- Multiple thin film barriers with different fluorides
  - Superior protection barrier
  - The right combination of fluorides
    - Can create "dielectric mirrors"
    - Able to reflect between 91 and 105 nm independent from aluminum

http://www.samaterials.com/fluorine/1227-magnesium-fluoride-mgf2-powder.html

http://www.samaterials.com/lithium/1209-lithium-fluoride-lif.html

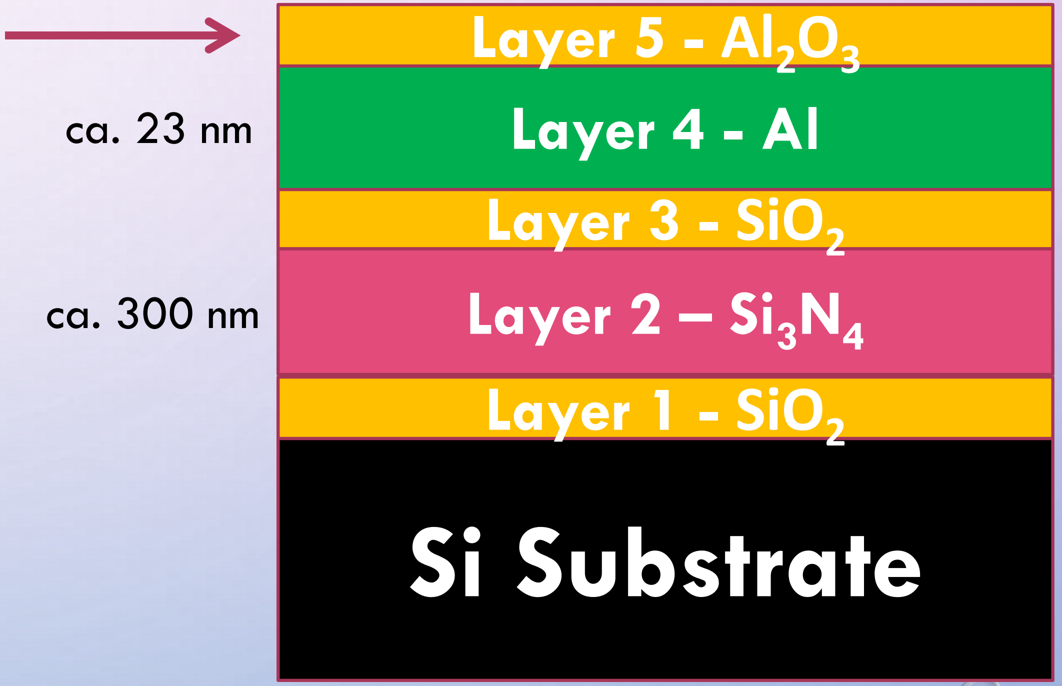
http://www.samaterials.com/fluorine/1375-sodium-aluminum-fluoride-na3alf6-cryolite.html

Dr. Matthew Linford/Dr. David Allred Labs

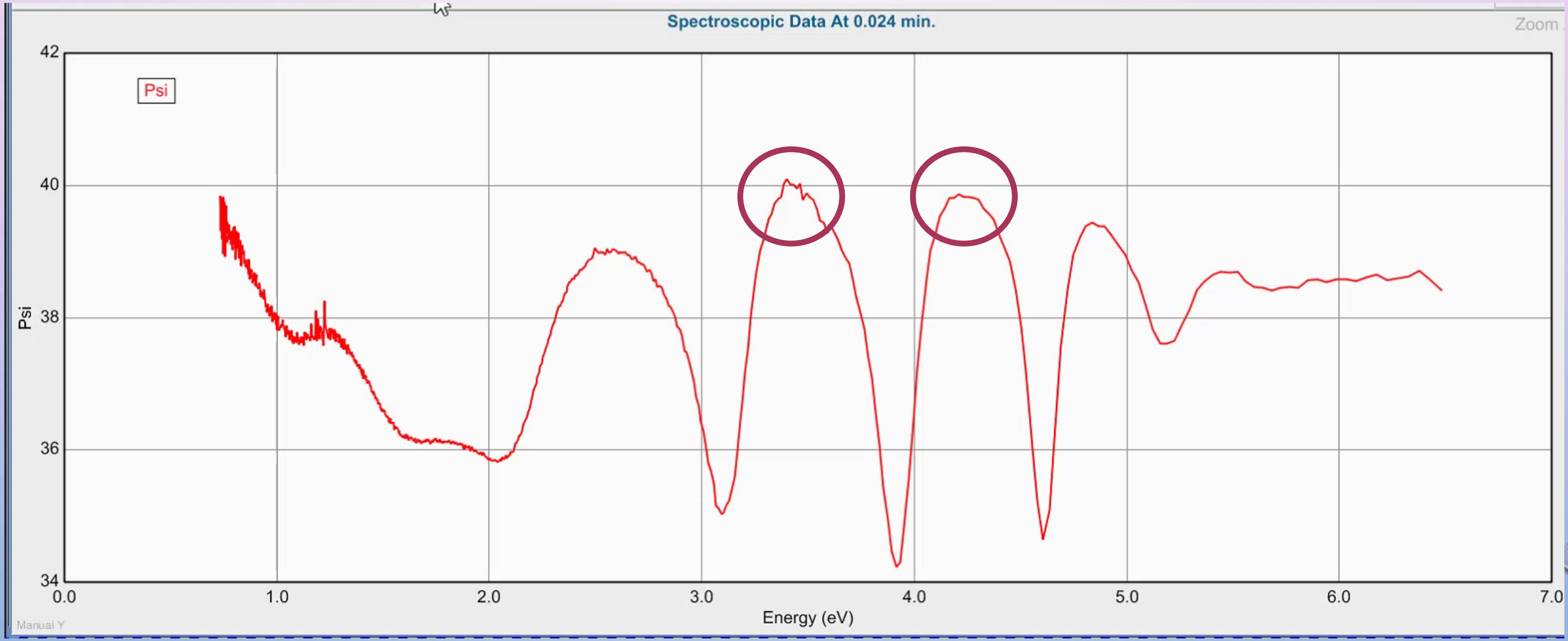
- End Goal
  - Dielectric mirror
  - Deposit and analyze
- Multiple thin film barriers with different fluorides
  - Superior Al protection barrier
  - The right combination of fluorides
    - Can create "dielectric mirrors"
    - Able to reflect between 91 and 105 nm independent from aluminum
- Develop/Refine analytical techniques

First Step-Oxidation of Bare Al

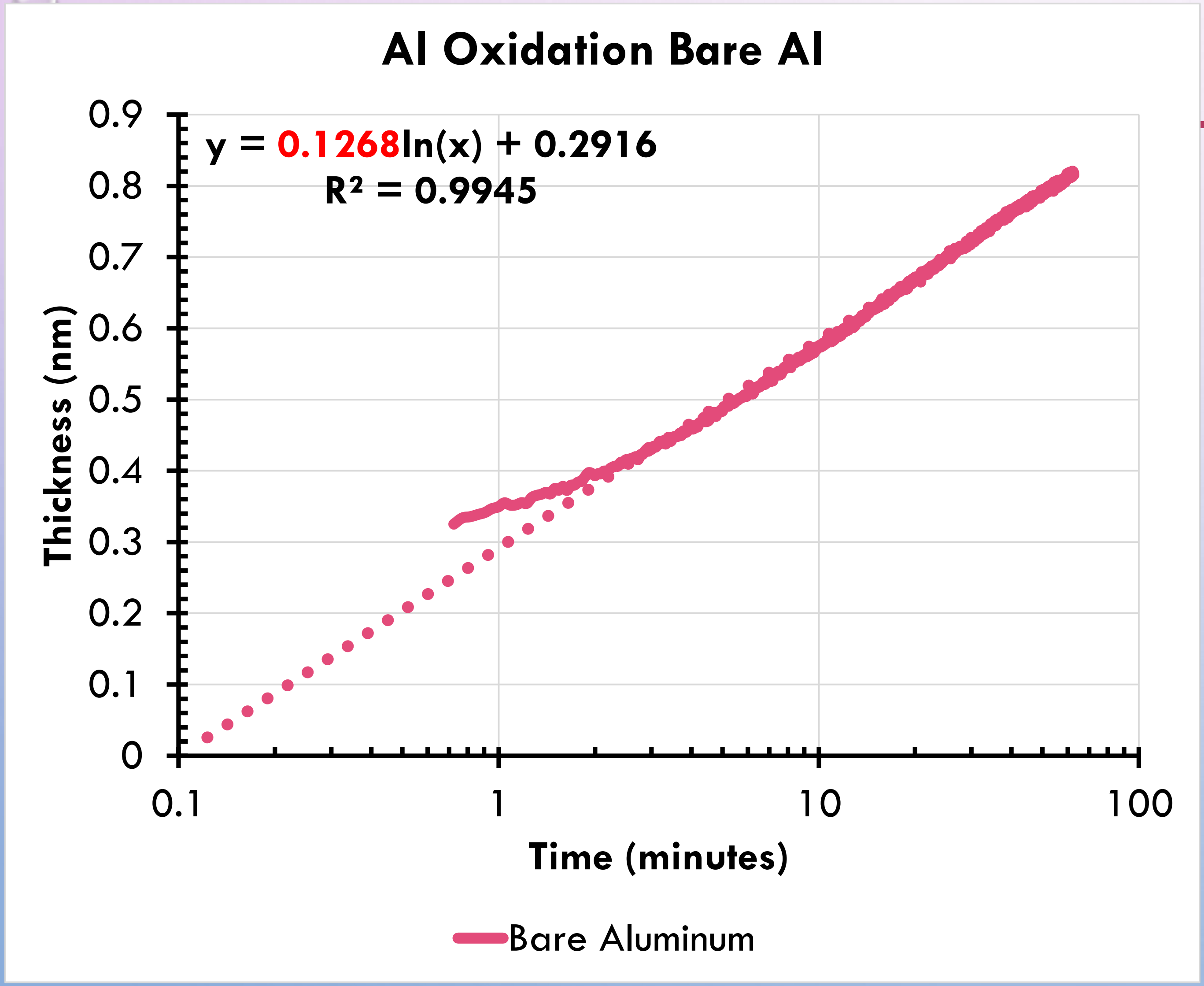
- Kurt J Lesker PVD 75
- JA Woolam SE
  - 5 feet apart
  - Dynamic mode

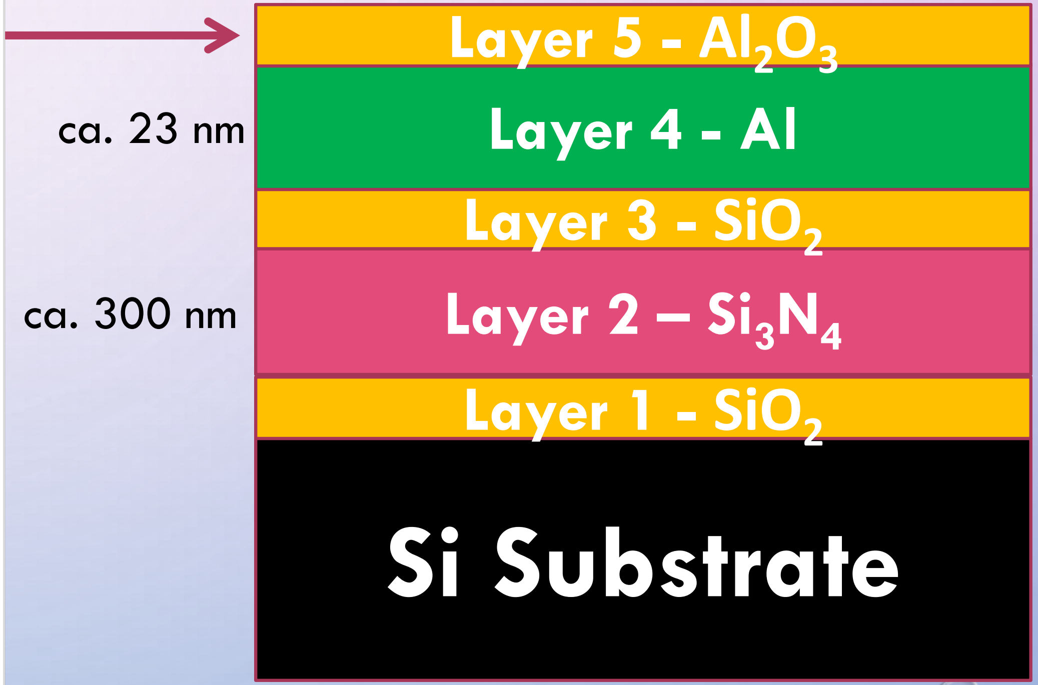


First Step-Oxidation of Bare Al



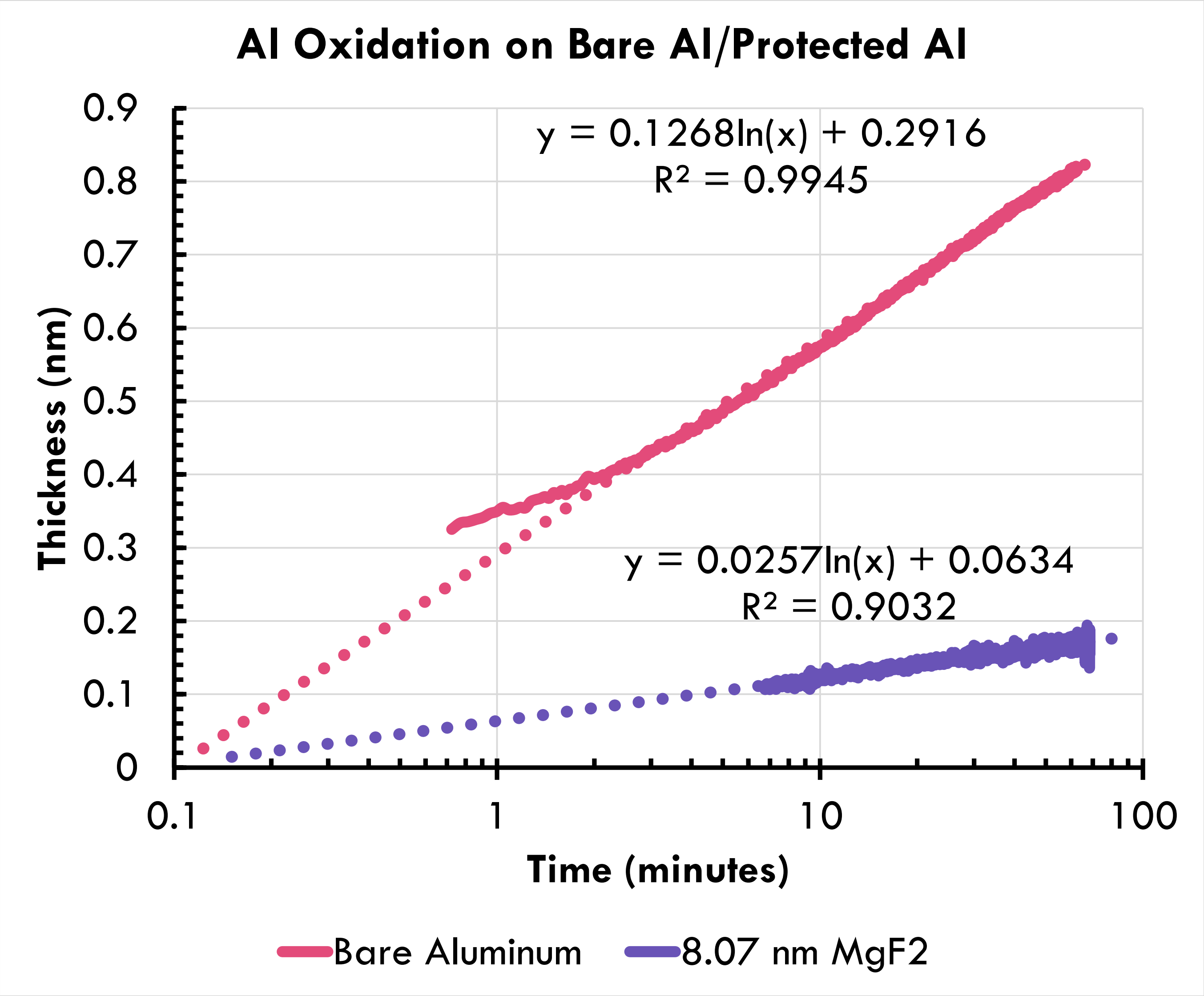
First Step-Oxidation of Bare Al

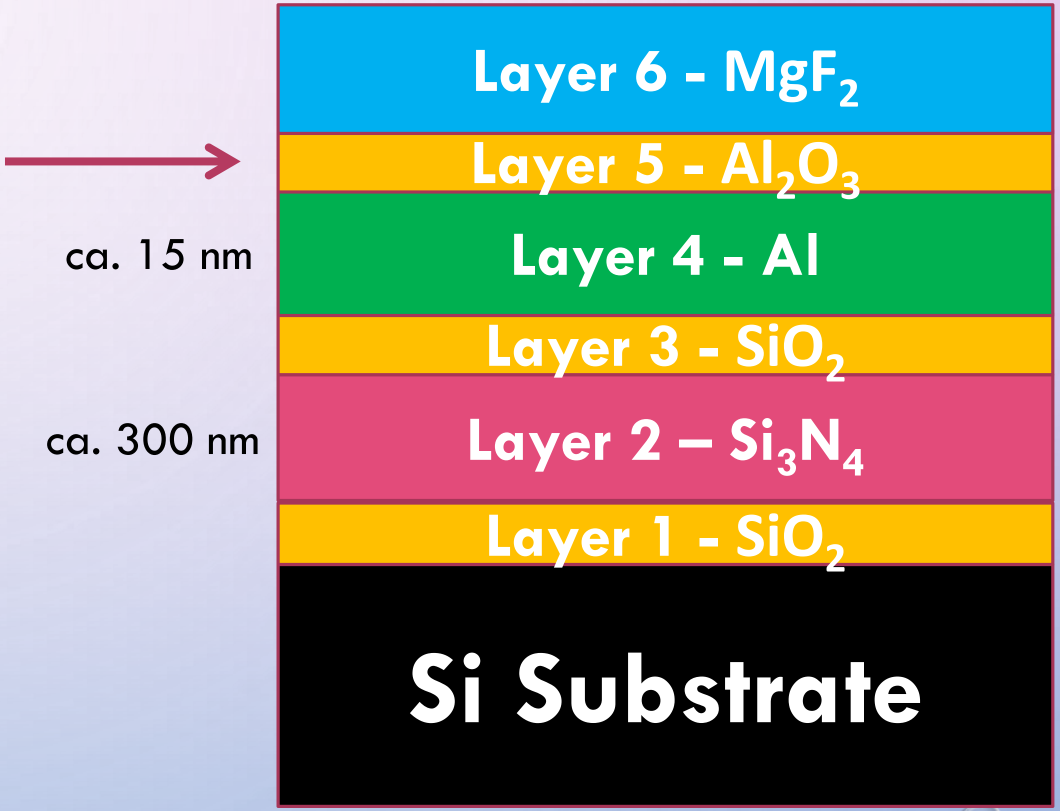




MSE-3.901

Oxidation of Protected Al





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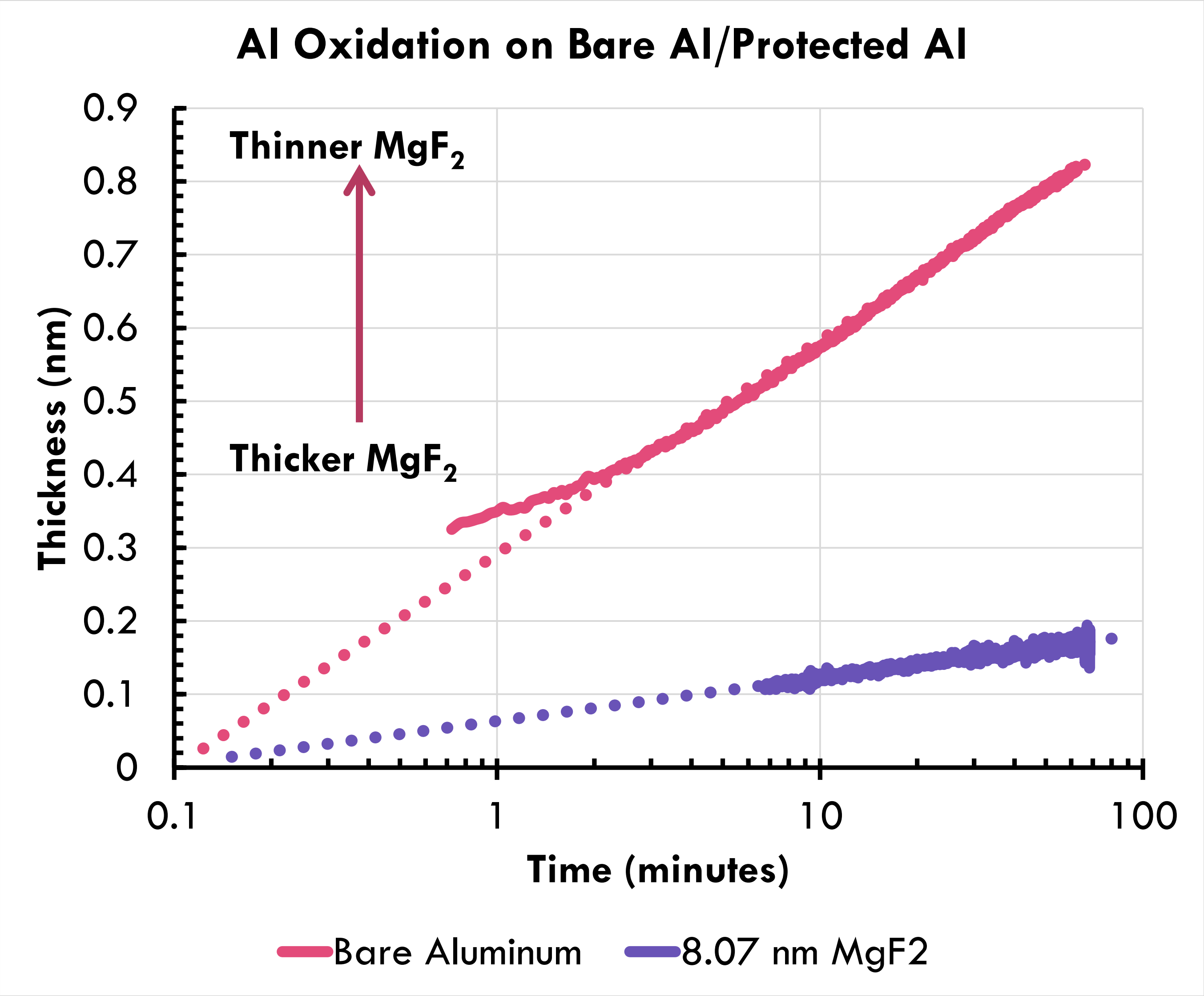

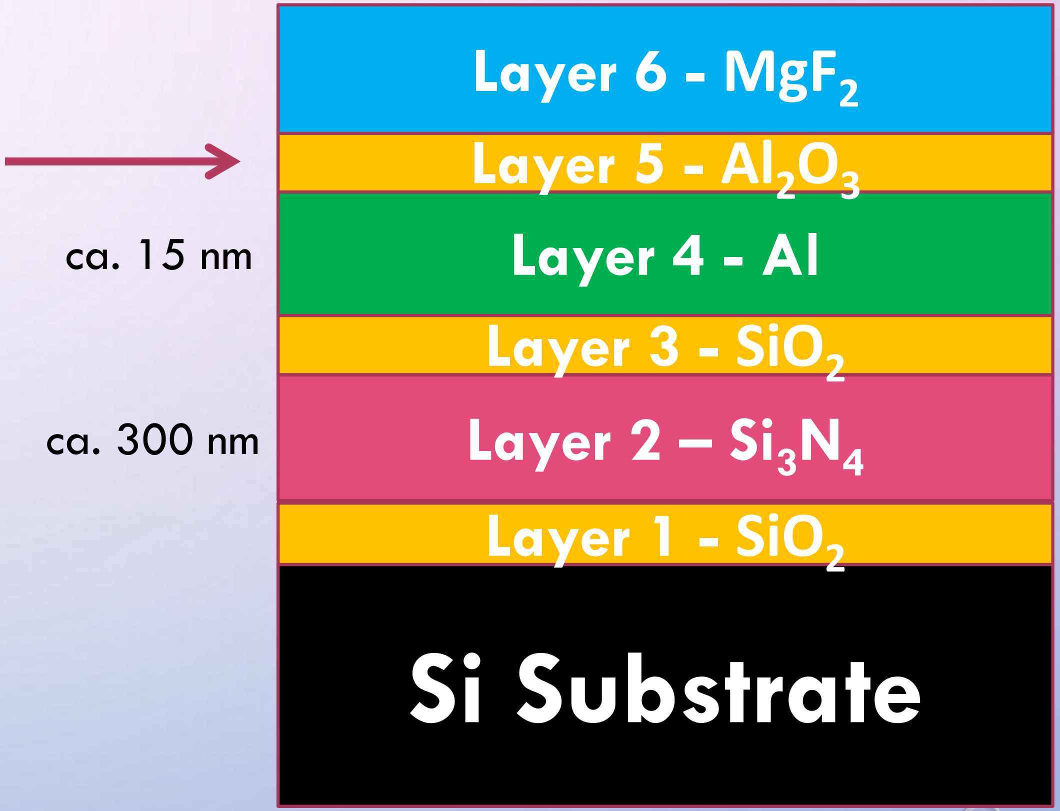

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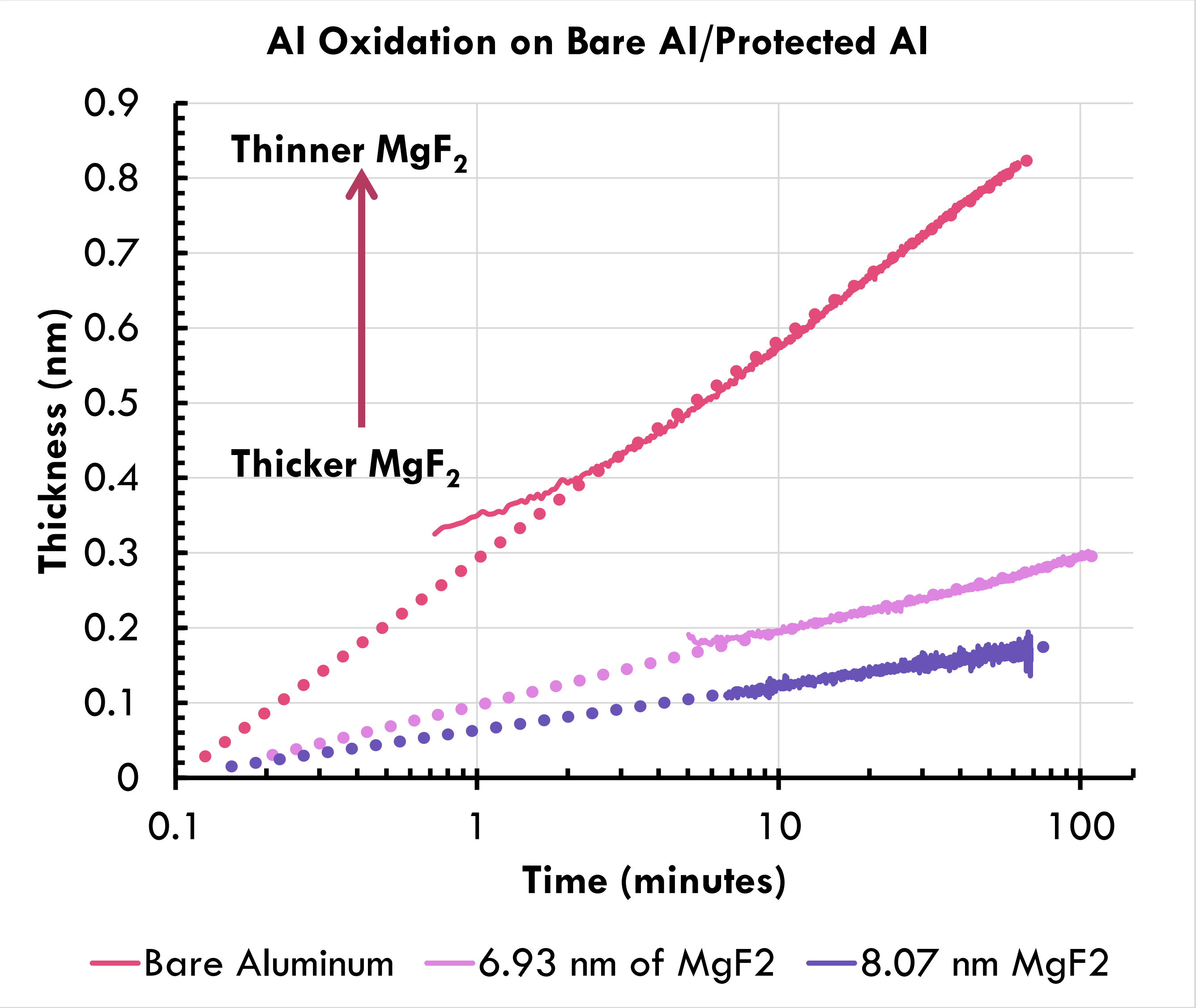

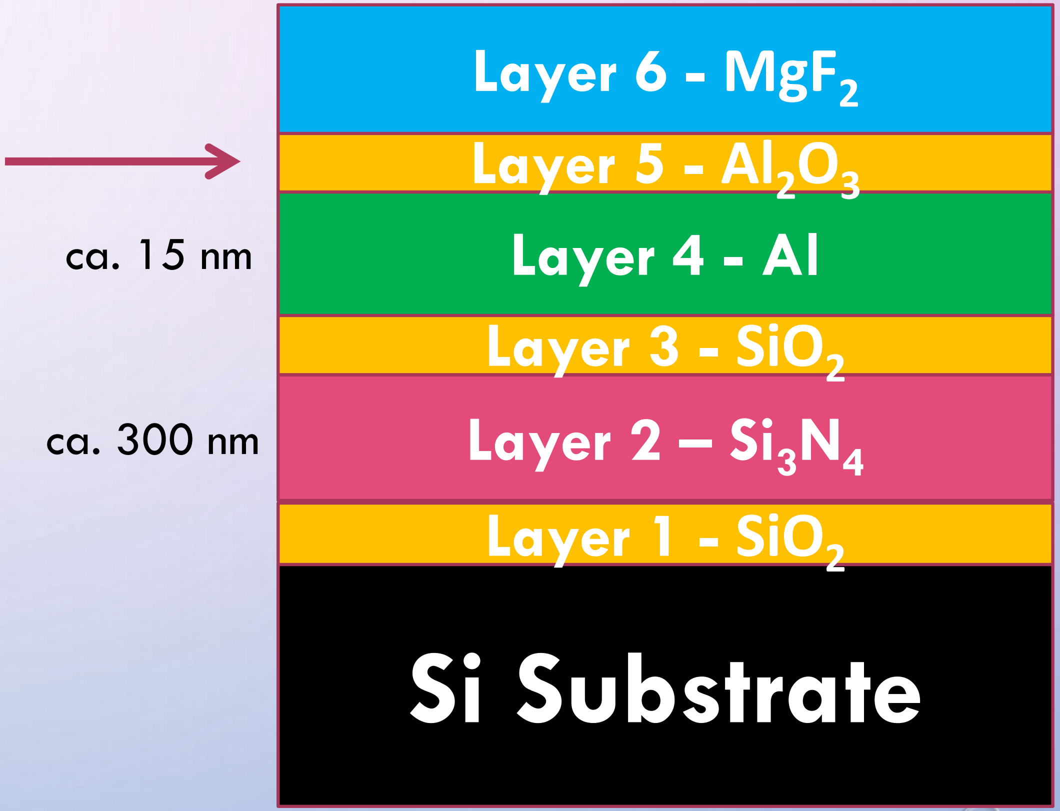

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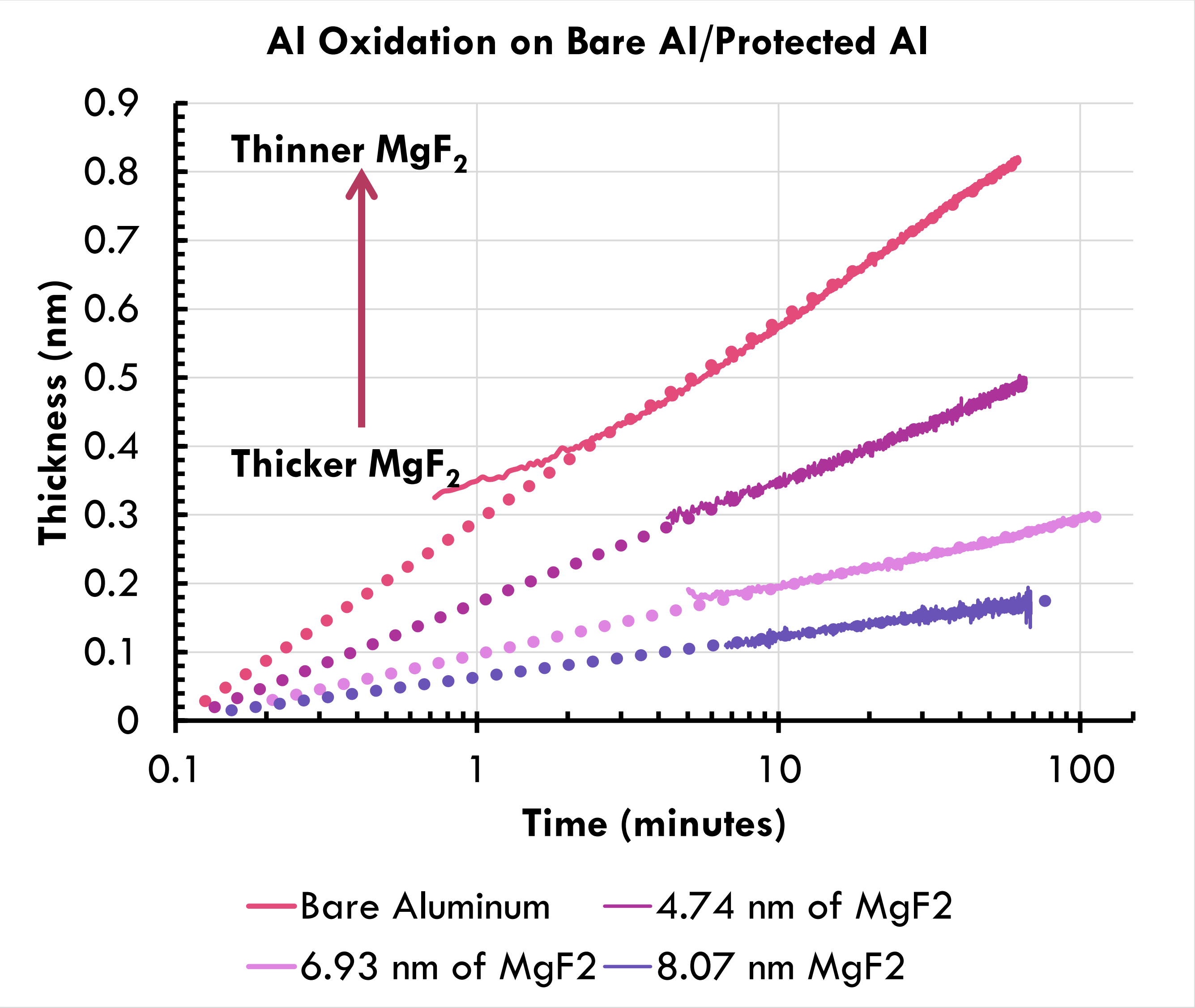

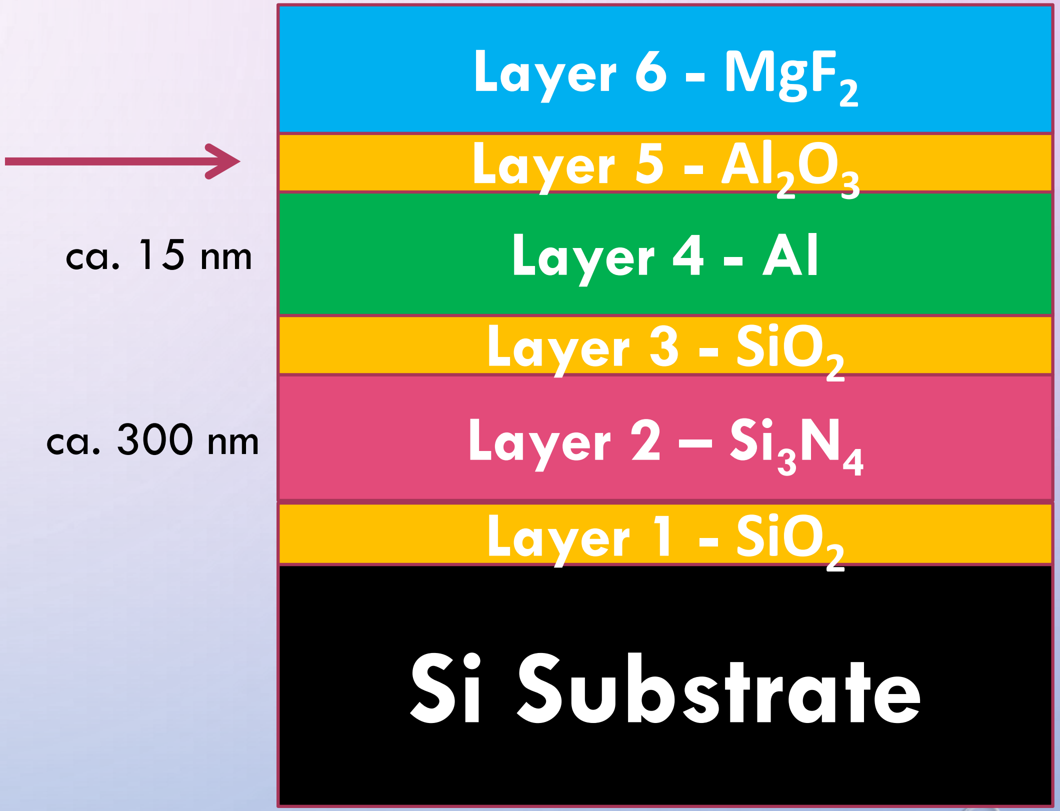

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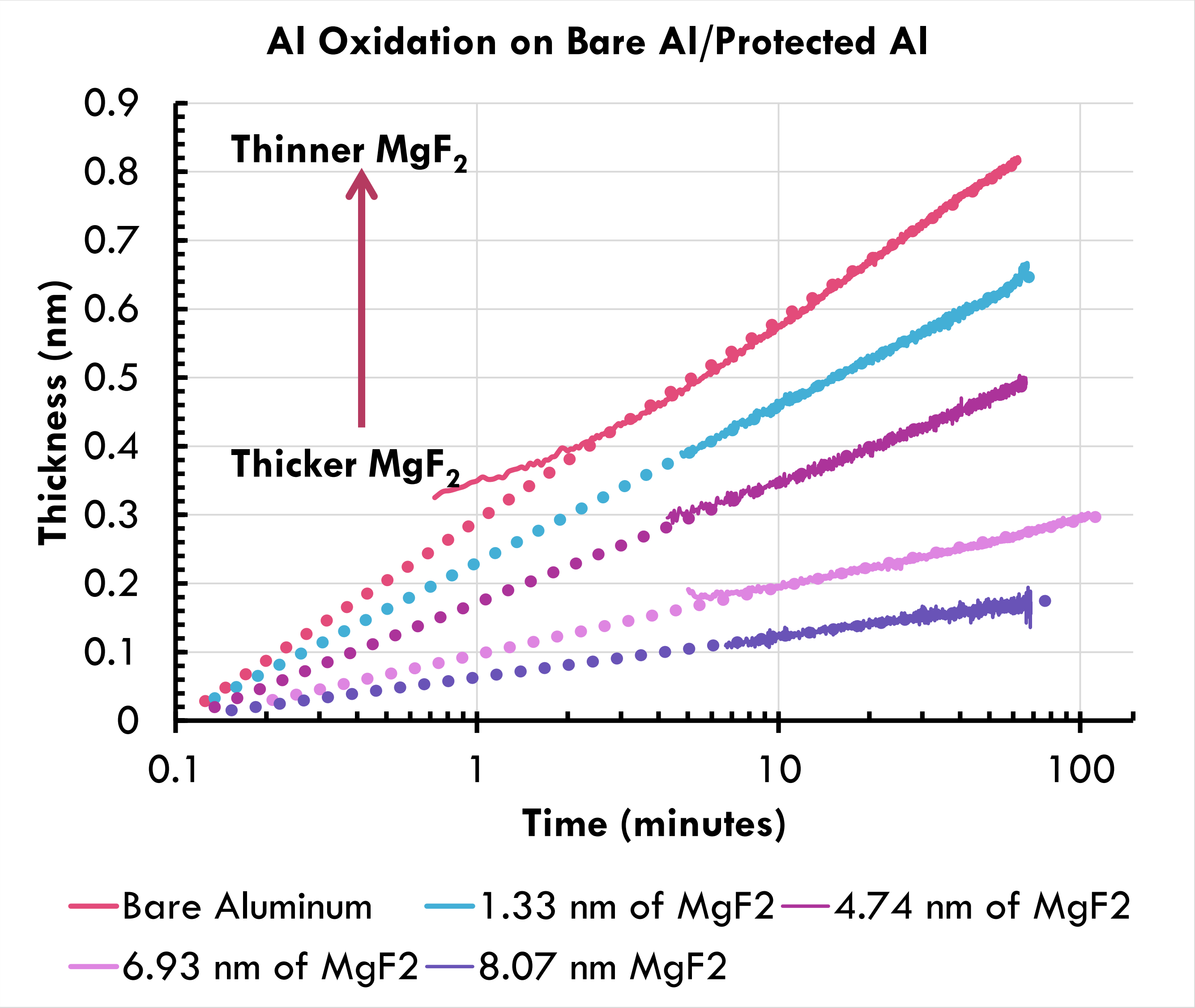

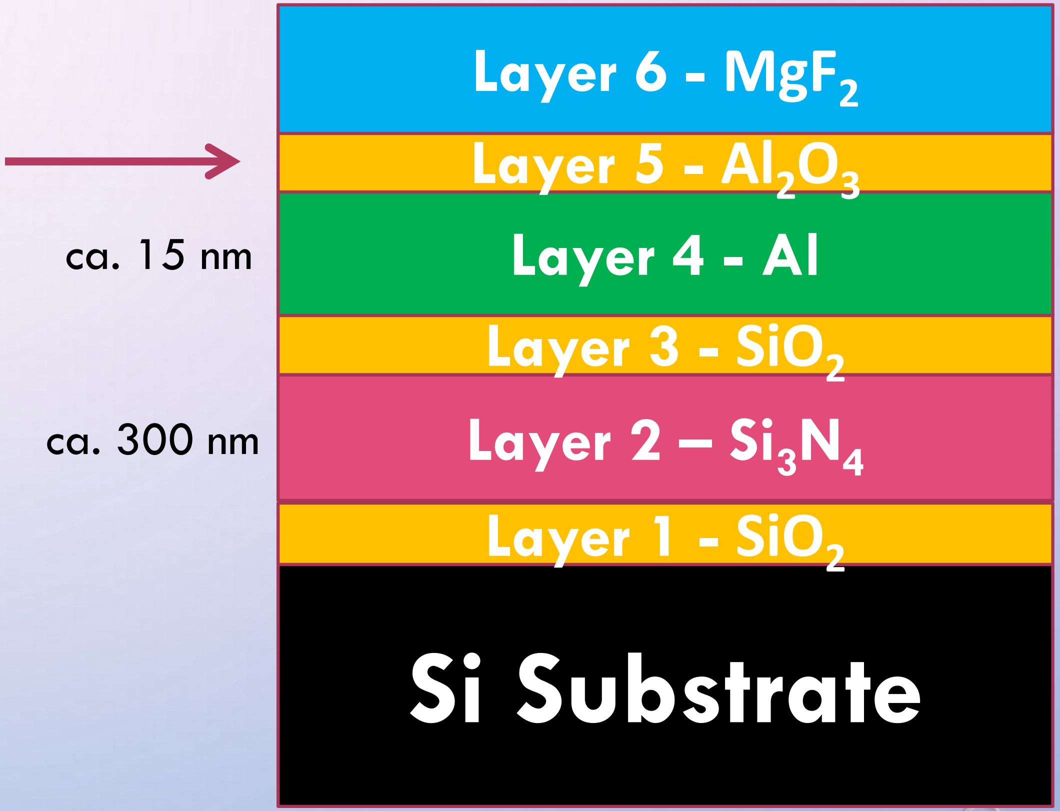

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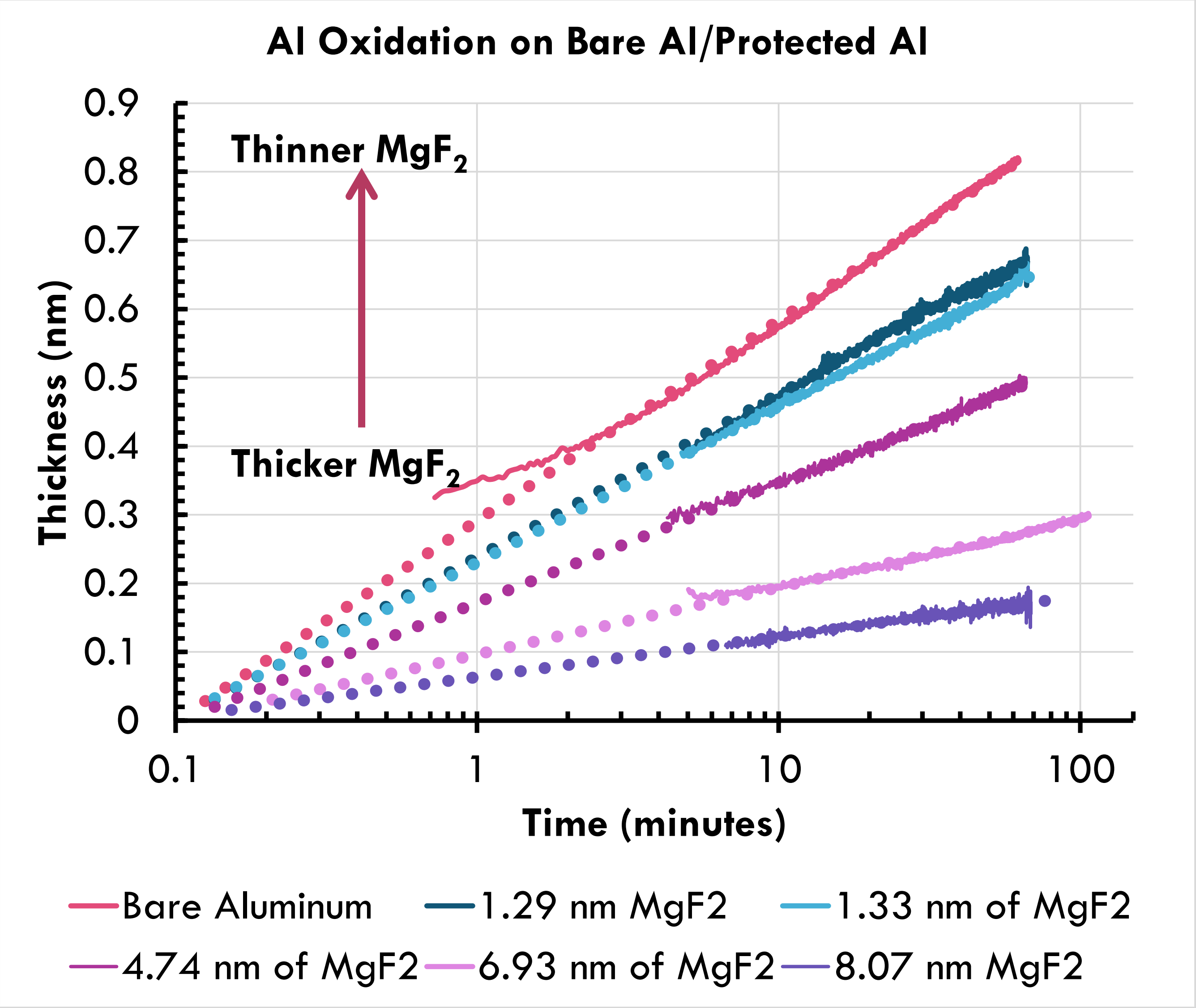

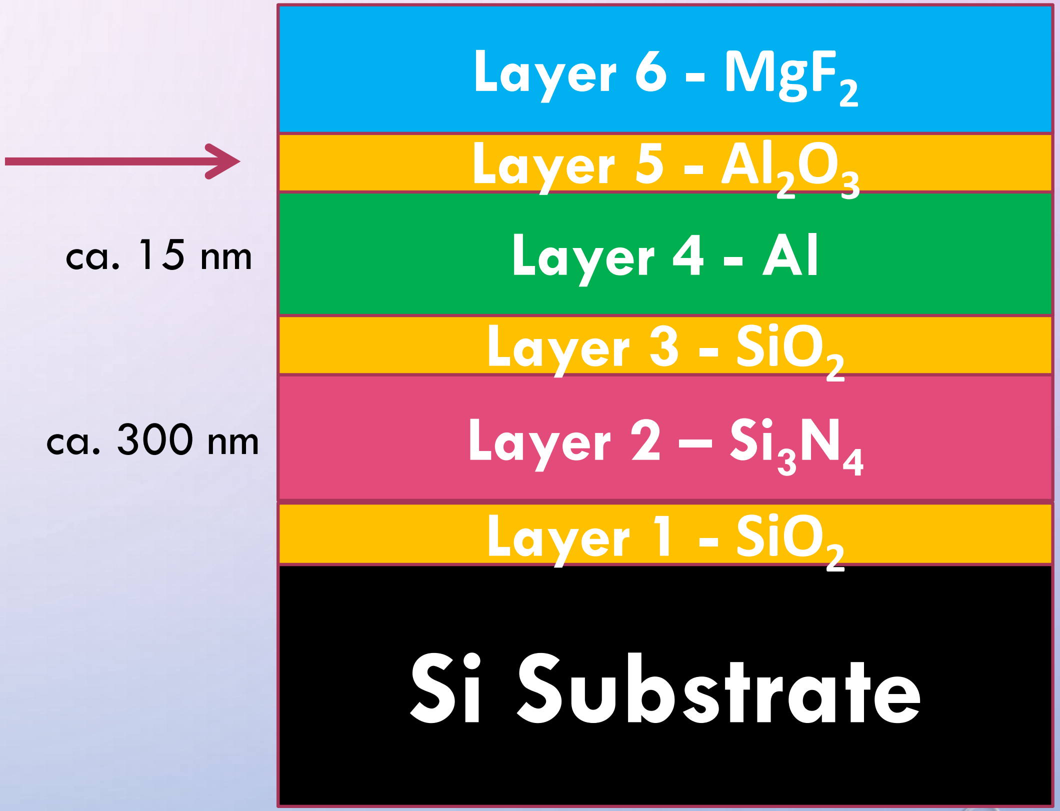

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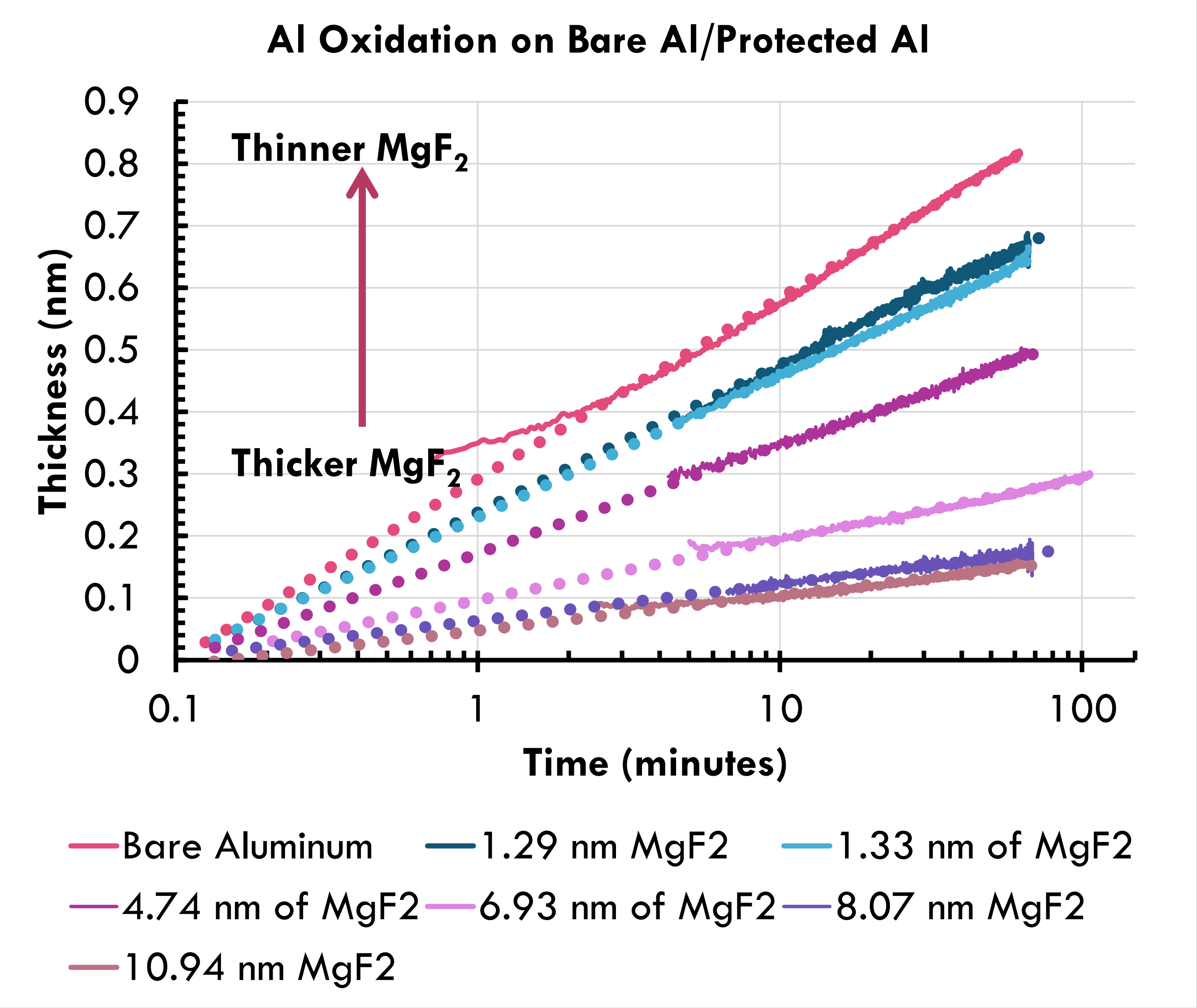
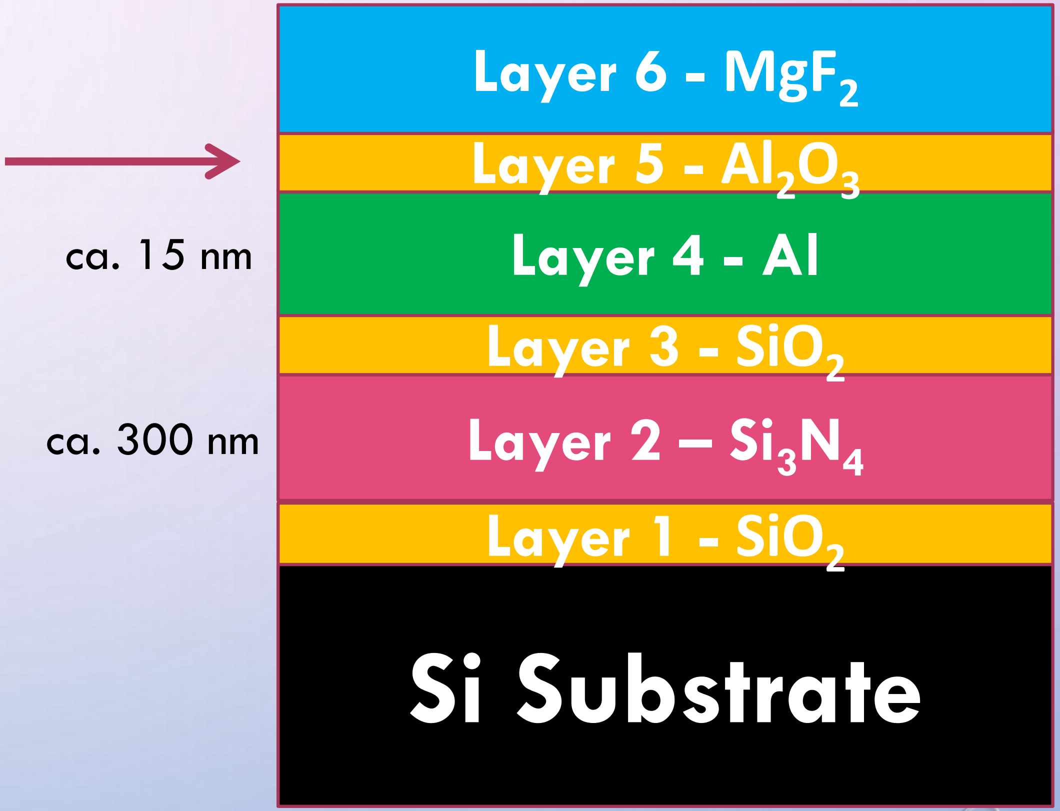
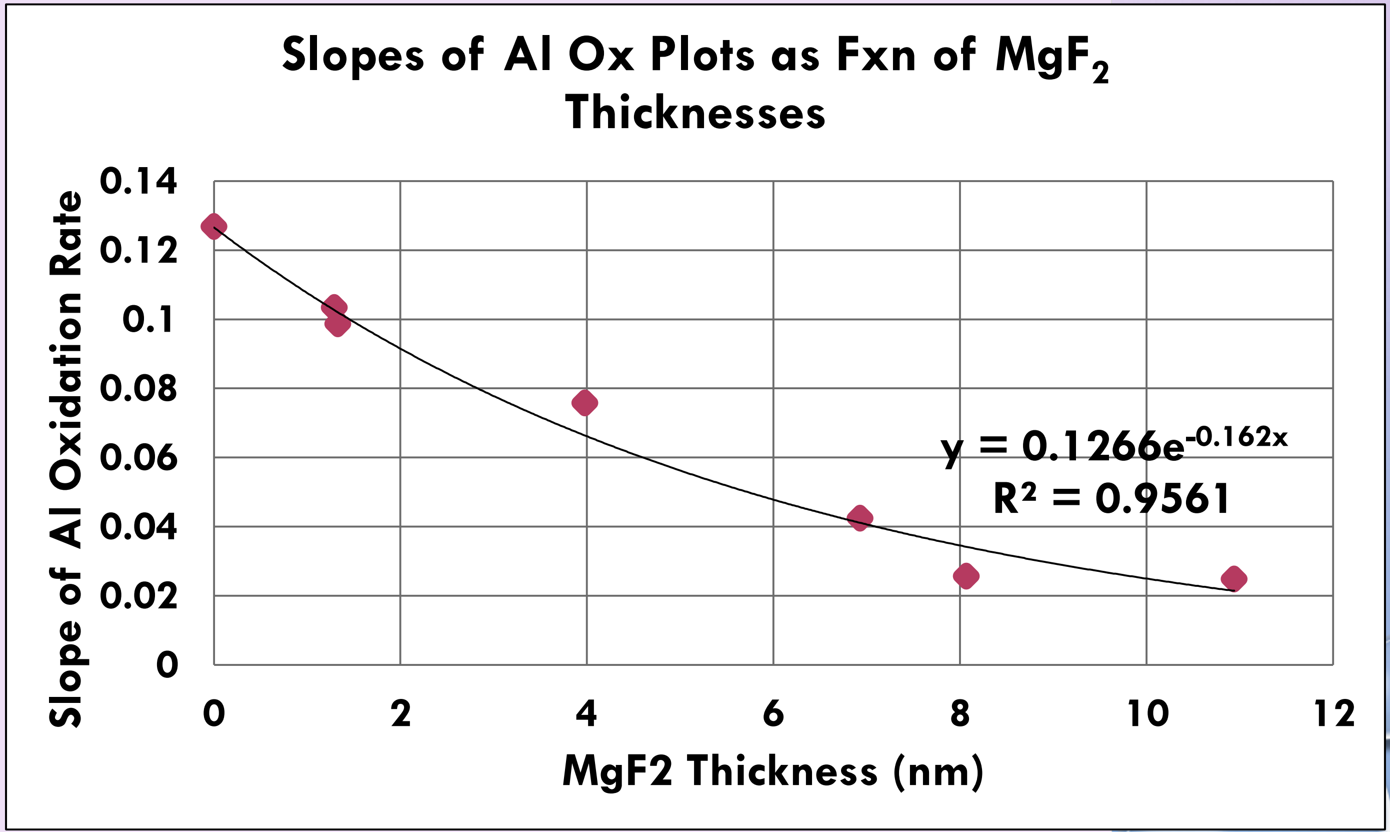




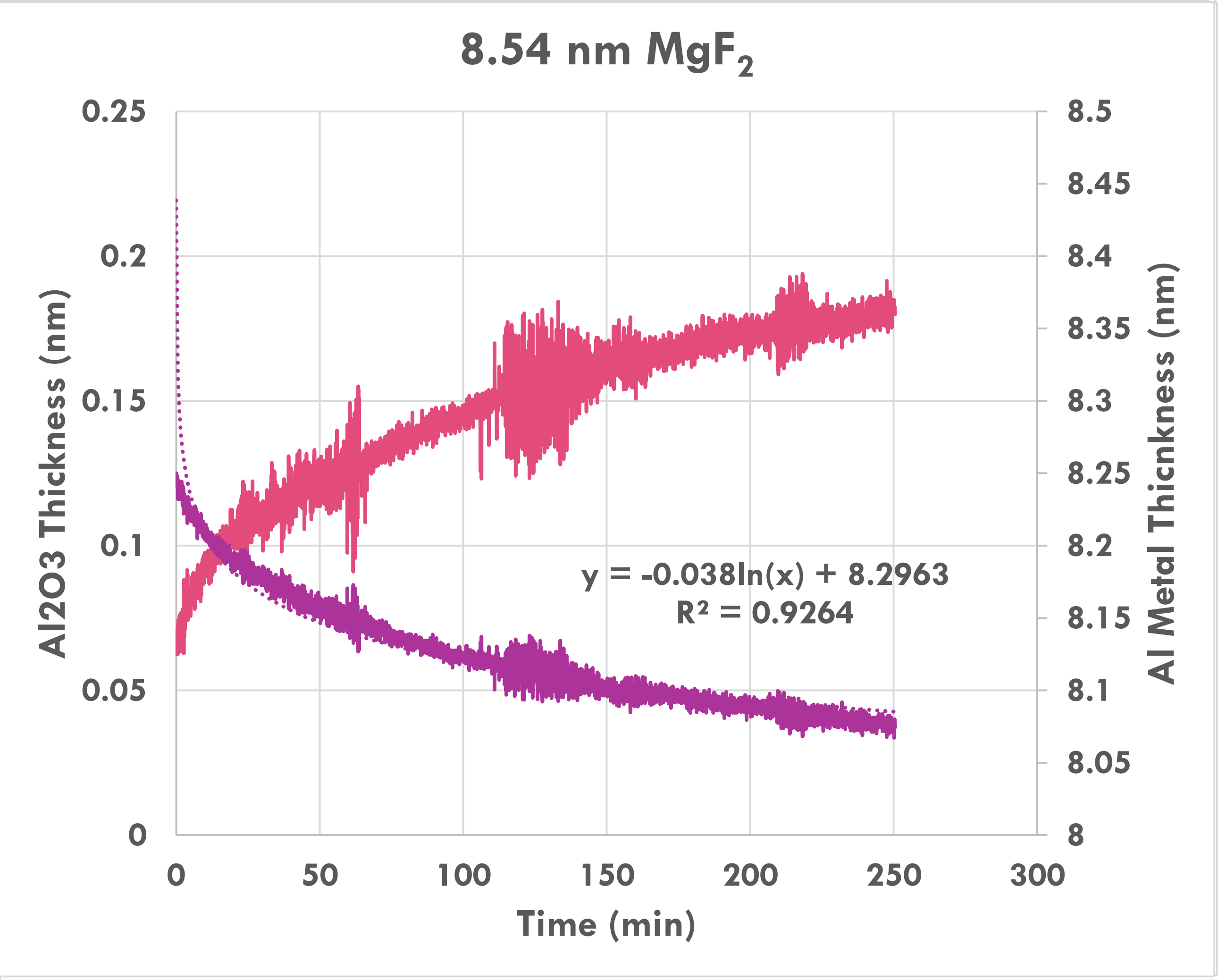
Oxidation of Al

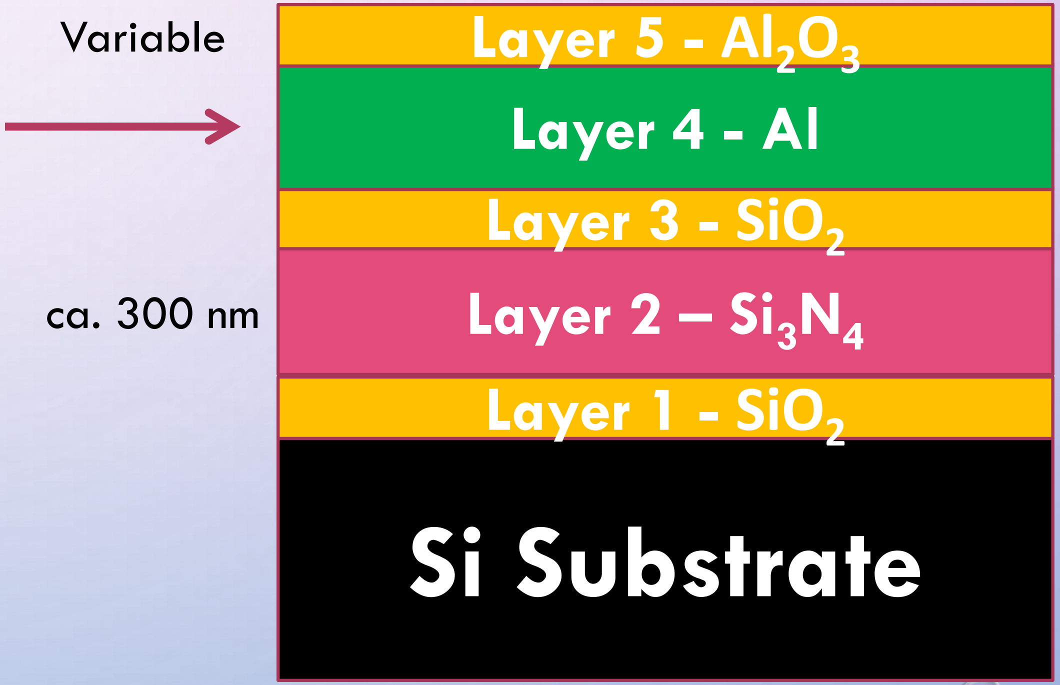
Slope of Oxidation Plots as a function of MgF_2 thickness

MgF_2 Thickness	Oxidation Slope
0	0.1268
1.29	0.1034
1.33	0.0987
3.98	0.0758
6.93	0.0424
8.07	0.0257
10.94	0.0248

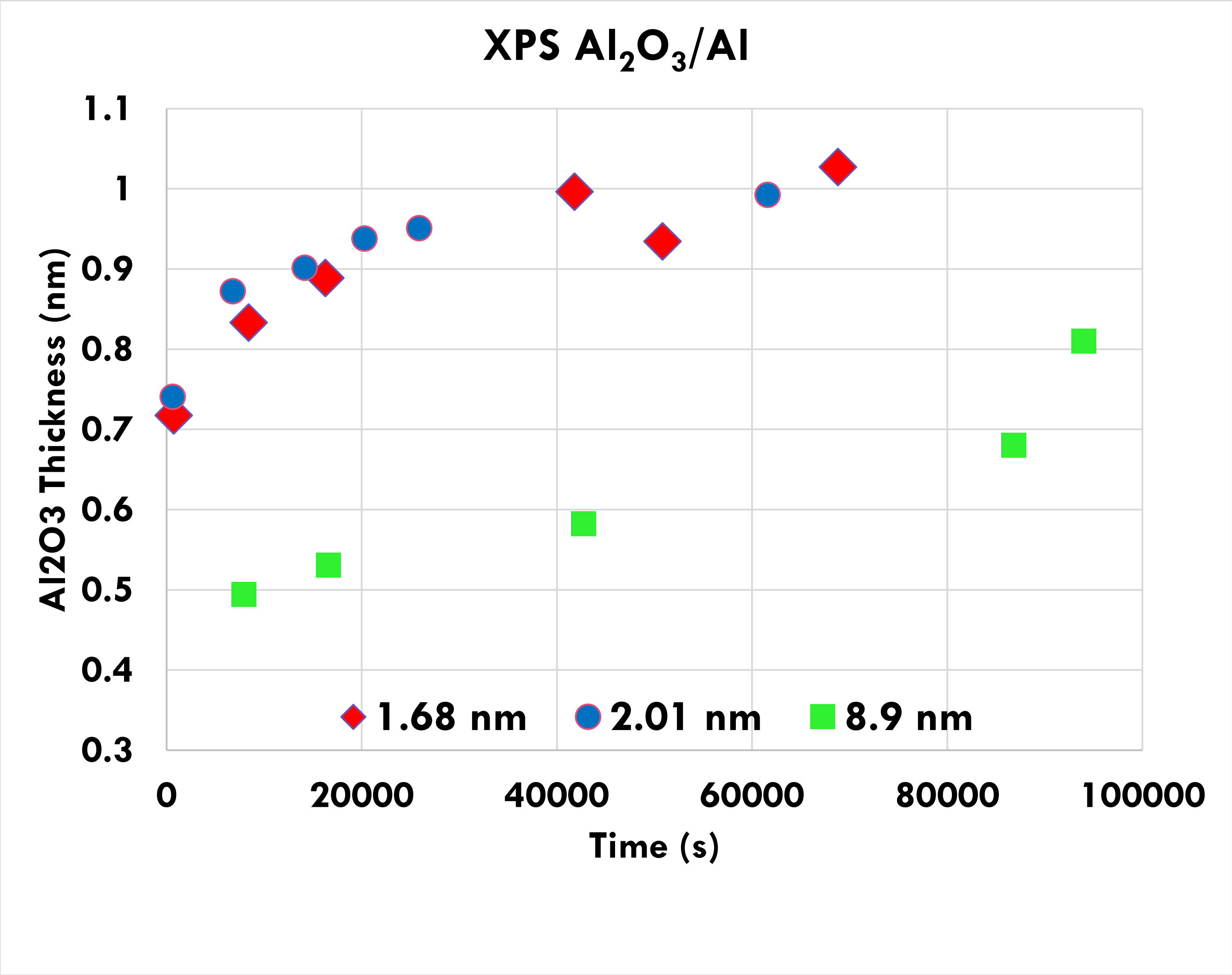


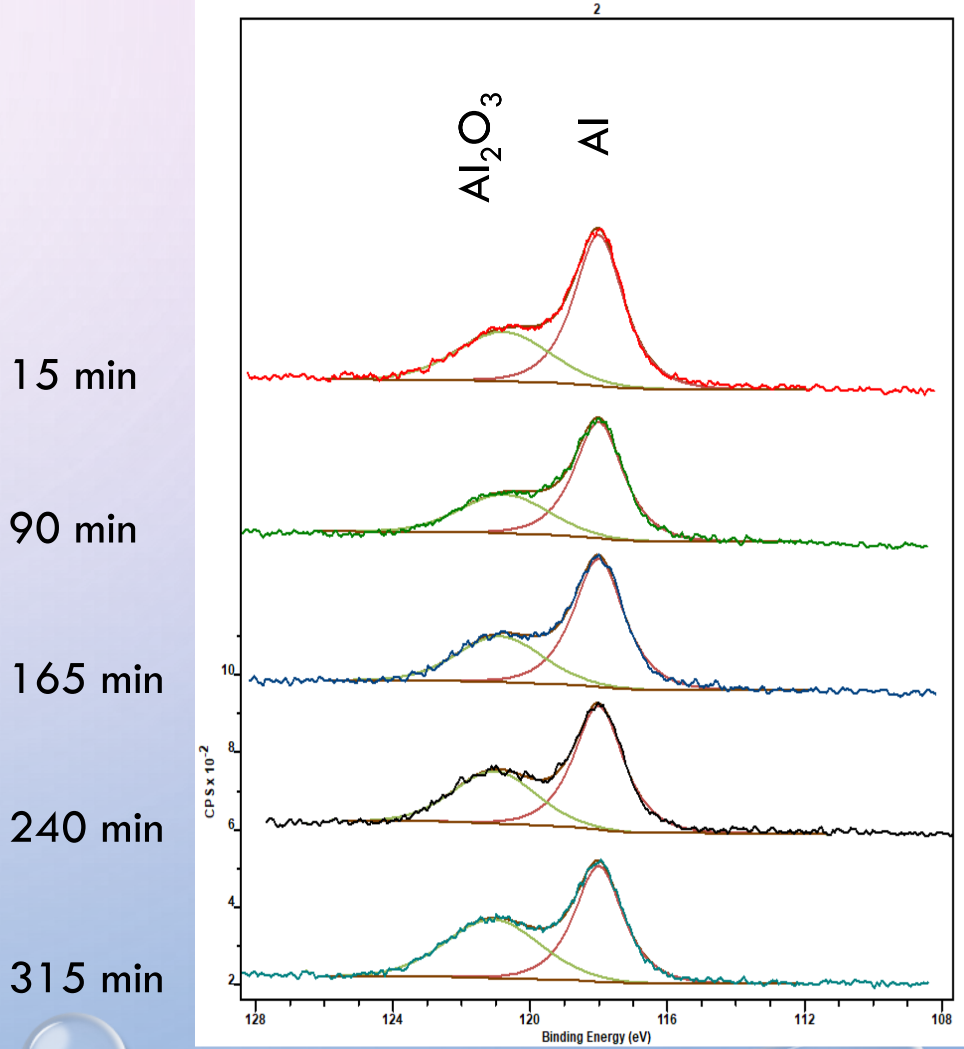
Decrease of Al Thickness





XPS


XPS Al2O3/Al
Al2O3 Thickness (nm)
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0
20000
40000
60000
80000
100000
Time (s)
1.68 nm
2.01 nm
8.9 nm


15 min
90 min
165 min
240 min
315 min
Al2O3
Al
2
CPS x 10-2
10
8
6
4
2
128
124
120
116
112
108
Binding Energy (eV)

Conclusion

- Analyze Al Oxidation
  - Bare Al
  - Protected Al
- Use of dynamic SE
  - Iterate SE models
    - Make consistent starting thicknesses with the Al_2O_3 and MgF_2
  - Plot oxidation slopes as functions of MgF_2 thickness
- XPS
  - Followed SE trends
  - Showed changes in Al oxidation state

- **Acknowledgements**
  - **Linford group**
    - **Cody Cushman, Tahereh gholian-Avval, Matthew Linford**
  - **Dr. Allred**
- **Brigham Young University**
- **NASA**

QUESTIONS?

WHAT WE ARE DOING

- **Understanding surface oxidation rates**
  - **Bare Al air oxidation**
- **Kurt J Lesker PVD 75**
  - **Sputtered Al**
    - **18-23 nm on Si_3N_4**
  - **Sample immediately placed on SE (<45 seconds)**
  - **Ex situ dynamic data collection in air for 1h (1.8 s)**
  - **Build up of Al_2O_3 with time**
  - **Al metal thickness decreases**

FIRST STEPS

**-TWO FITS

STATIC

DYNAMIC

1-PUT THE AL2O3 TO 0 TO 0.1 NM

2-FIX THE MGF2 THICKNESS TO WHAT THE QCM INDICATED

3-FIX THE AL THICKNESS TO WHAT THE QCM INDICATED

4-USE AL LORENTZ MODEL

5-ADJUST THE COEFFICIENTS FOR THE PARAMETERS IN THE STANDARD WOOLLAM AL LORENTZ MODEL-TO GET LOW MSE

6-FIT AL THICKNESS

7-RUN DYNAMIC FIT

8-USE DYNAMIC DATA TO SET THICKNESS OF AL2O3 TO 0 AT TIME 0

9-FIX THE AL2O3 THICKNESS IN THE MODEL TO THIS POINT

10-ITERATE IN ORDER TO REDUCE MSE AND BE CONSISTENT WITH THE EVAPORATION

FIRST STEPS

Static Fit

Fix Al_2O_3 to 0.1 nm

Fix MgF_2 and Al to QCM values

Relax Al coefficients

Low MSE

Dynamic fit

Iterate $Al_2O_3/MgF_2/Al$

Low MSE

Now what is it that we can see from the dynamic fit that helps us?