200203 - PdAg + 550 K O2 + H2

Monday, February 3, 2020 9:39 AM

Plan

Test reproducibility of 550 K 5 min oxidation and establish protocol

Logbook

Legend - Plan in purple

Flash anneal to 800 K
Set up water cooling
Open gate valve

Measure clean Ag and turn off O3 (8:50 AM)
XPS: clean Ag - 66 degrees
Switch on evap
Connect cooling
9:45 AM

- **Pd evaporation**: 2.50 A, 1000 V, 10.0 mA, 1.7-2.0 nA for **1 min 20 sec** at 300K
  Goal: 0.10 ML
  0.14/1.5 = 0.0933
  0.1/0.0933 = 1.0718
  1.0718*2.0/1.7 = 1.2609
  0.26*60 = 15.6

Switch on X-Ray source before transfer

XPS: Pd deposition - 0 degrees
- Pd area: 11 kcps eV
- Ag area: 713 kcps eV
- Pd/Ag: 11/713 = 0.0154 --> **0.09(3) ML GREAT**

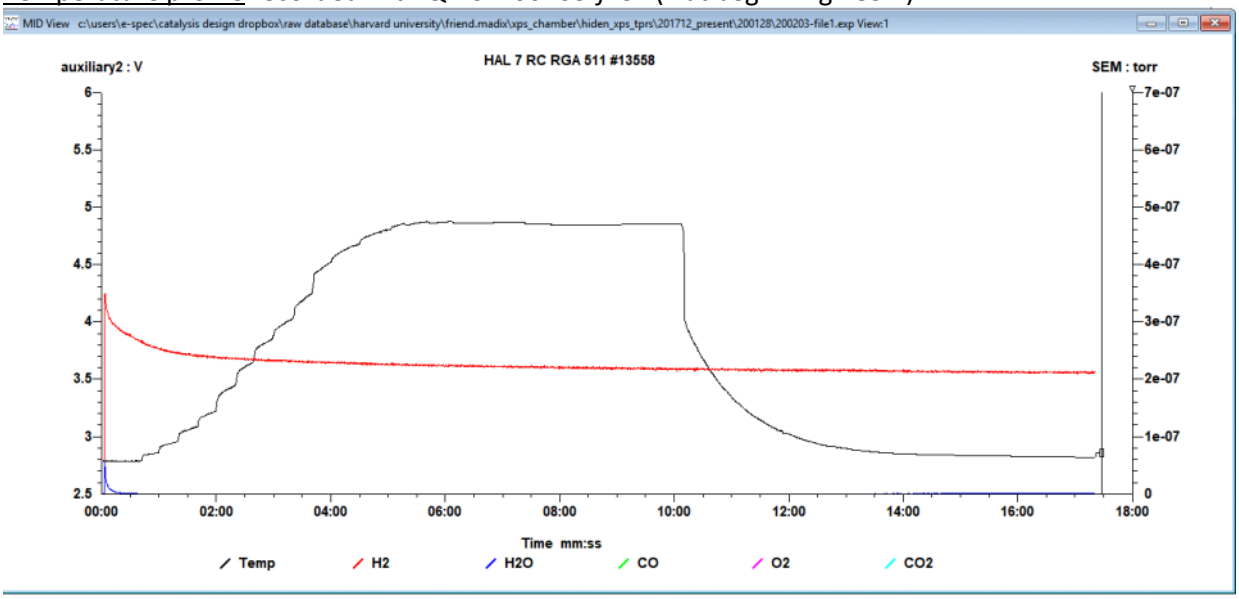
XPS: Pd deposition - 66 degrees
- Pd area: 11 kcps eV
- Ag area: 275 kcps eV
- Pd/Ag: 11/275 = 0.04 --> **0.10(8) ML TOP**

Done at 10:15 AM

Turn up O2 at 10:30 AM

- **Oxidized** in 1.92(7) Torr O2 at 550 K for **5 min**
  - Valve cracked
  - O2 flowing from O3 conditions before
  - turbo 19 W
  - 1, 3, 5, 7, 10, 12, 15, 17, 20, 23, 24, 25 W to ramp (in 20 sec intervals); let climb to 550 K at 25 W (started timer when it hit 550 K); 23.5 W laser power to stabilize at 550 K
  - maintained T within 550-554 K over duration of oxidation
  - Cooling in O2 till T = 310 K (~1 min) , then evacuating HP cell till p_baratron = 0.1 mTorr 10:55 AM

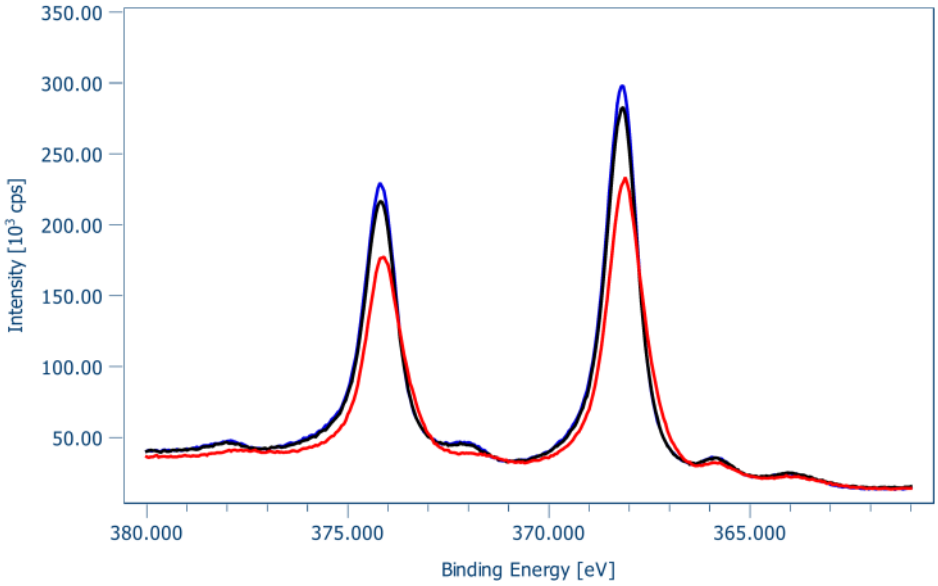
Temperature profile recorded with QMS: *200203-file1* (T at beginning 285 K)



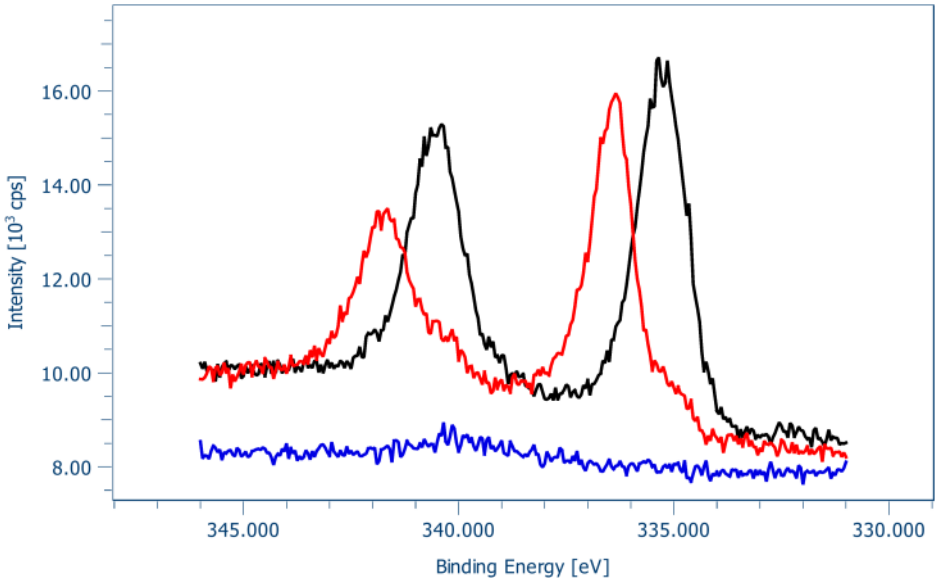
Switch back to air cooling for H2 dose; clear water from cryostat
Shut valve to HP cell at 15.0"
Turn on X-ray source at 15.0"

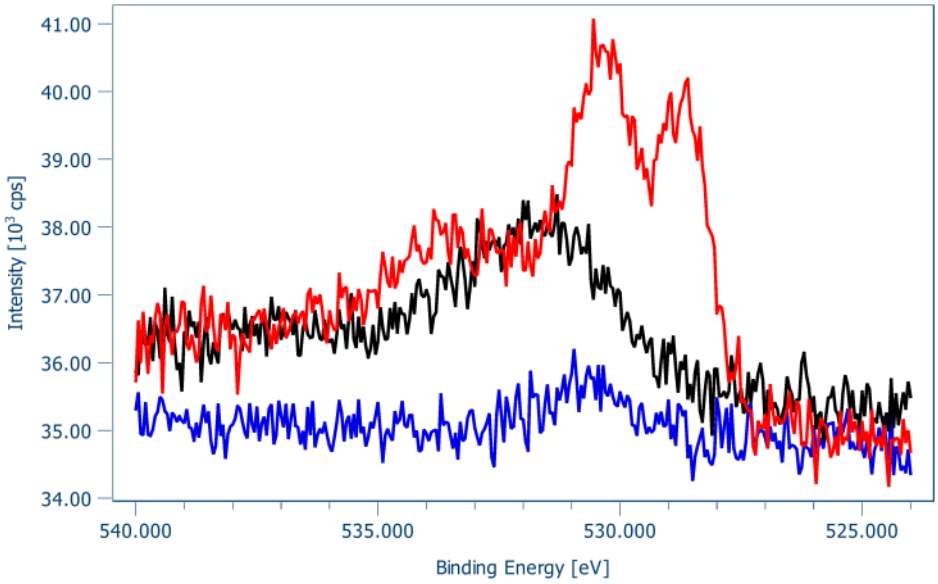
O2 pre-stage pressure was 1925 psi at 11:15 AM

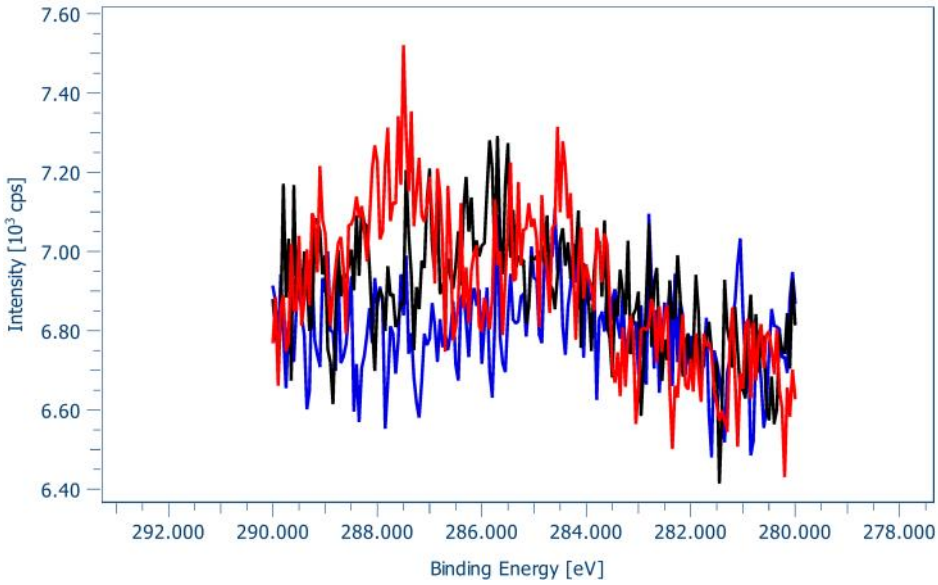
Measure
XPS: after oxidation - 66 degrees



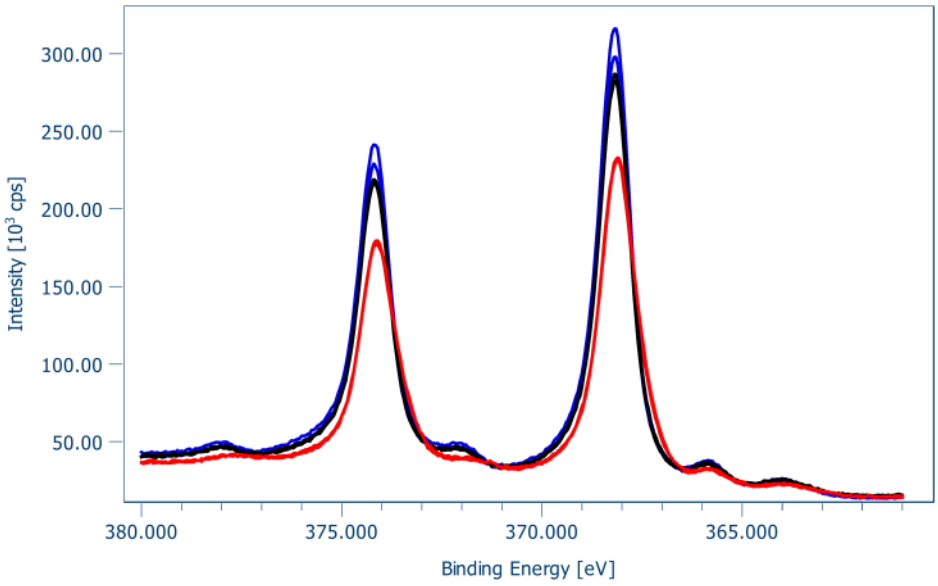
Pd metallic ~335.1-2 eV

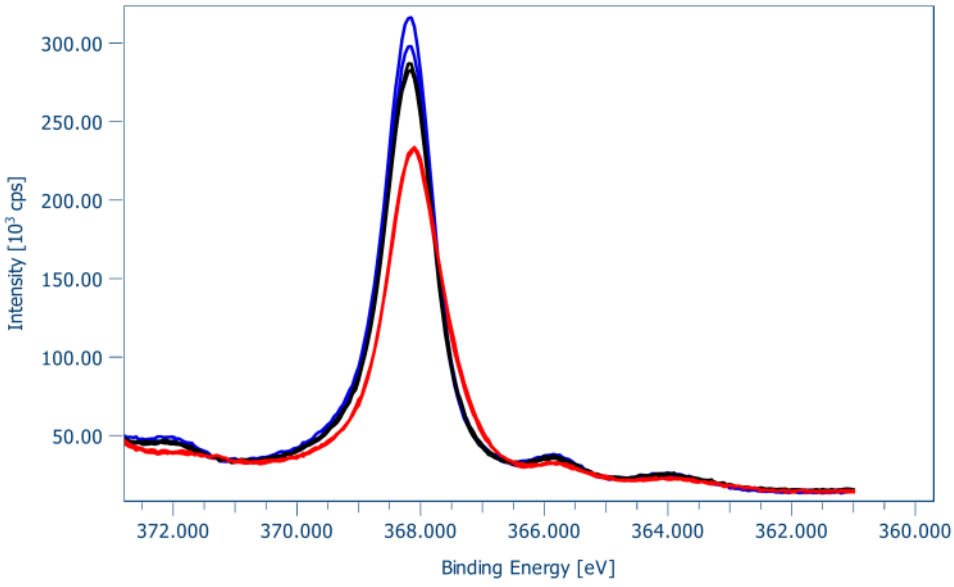

16.00
14.00
12.00
10.00
8.00
Intensity [10³ cps]
345.000
340.000
335.000
330.000
Binding Energy [eV]

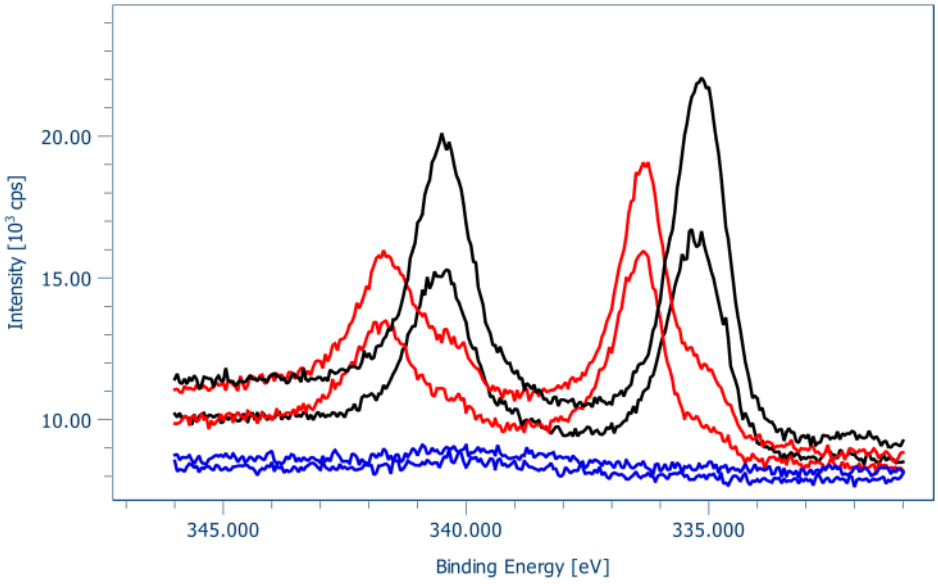

41.00
40.00
39.00
38.00
37.00
36.00
35.00
34.00
Intensity [10³ cps]
540.000
535.000
530.000
525.000
Binding Energy [eV]

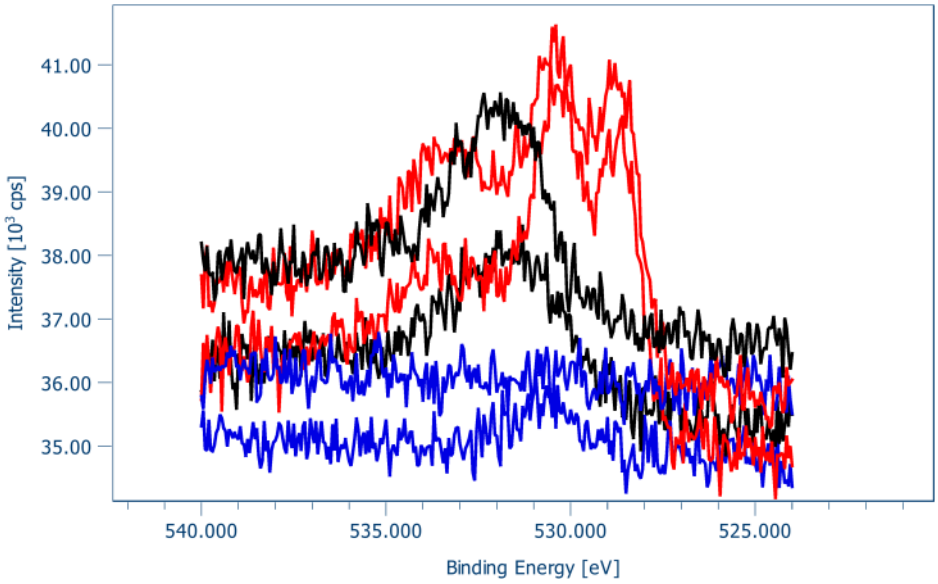

7.60
7.40
7.20
7.00
6.80
6.60
6.40
Intensity [10³ cps]
292.000
290.000
288.000
286.000
284.000
282.000
280.000
278.000
Binding Energy [eV]

Comparison to 200128 below: Fantastic!, oxidized (red) extremely similar
Reminder 200128 was 0.14 ML Pd!









30 mins exposure to background with X-ray gun on

XPS: 30 mins bkg - 66 degrees

Start sputter 12:12 AM
Start O3 12:10 AM

- Sputter for 45 mins at 20 mA and 3e-5 Torr Ar
- Anneal to 800 K for 10 min

##
2nd round
##

Leak O3 in HP cell 1:00 PM
Stop leak O3 in HP cell 2:05 PM

XPS: clean Ag#2 - 66 degrees
Switch on evap
Connect cooling
2:20 PM

- **Pd evaporation**: 2.50 A, 1000 V, 10.0 mA, 1.6-1.2 nA for **1 min 30 sec** at 300K
  Goal: 0.10 ML
  0.14/1.5 = 0.0933
  0.1/0.0933 = 1.0718
  1.0718*2.0/1.7 = 1.2609
  0.26*60 = 15.6

Switch on X-Ray source before transfer

XPS: Pd deposition#2 - 0 degrees
- Pd area: 10 kcps eV
- Ag area: 717 kcps eV
- Pd/Ag: 10/717 = 0.0139 --> **0.08(4) ML fine compared to deposition before**

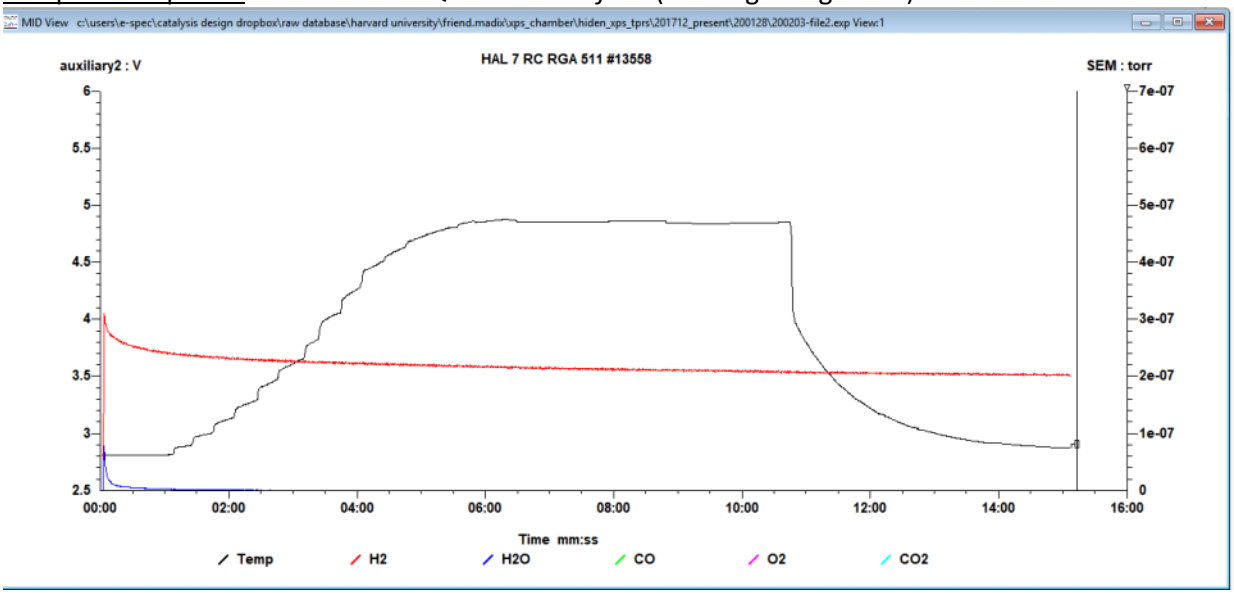
XPS: Pd deposition#2 - 66 degrees
- Pd area: 8.5 kcps eV
- Ag area: 280 kcps eV
- Pd/Ag: 8.5/280 = 0.0304 --> **0.08(3) ML very accurate angle at low coverages**

Done at 3:01 PM

Turn up O2 at 3:14 APM

- **Oxidized** in 1.92(6) Torr O2 at 550 K for **5 min**
  - Valve cracked
  - O2 flowing from O3 conditions before
  - turbo 19 W
  - 1, 3, 5, 7, 10, 12, 15, 17, 20, 23, 24, 25, 25.5 W to ramp (in 20 sec intervals); let climb to 550 K at 25.5 W (started timer when it hit 550 K); 23.5 W laser power to stabilize at 550 K
  - maintained T within 550-554 K over duration of oxidation
  - Cooling in O2 till T = 302 K (~1 min) , then evacuating HP cell till p_baratron = 0.1 mTorr 10:55 AM

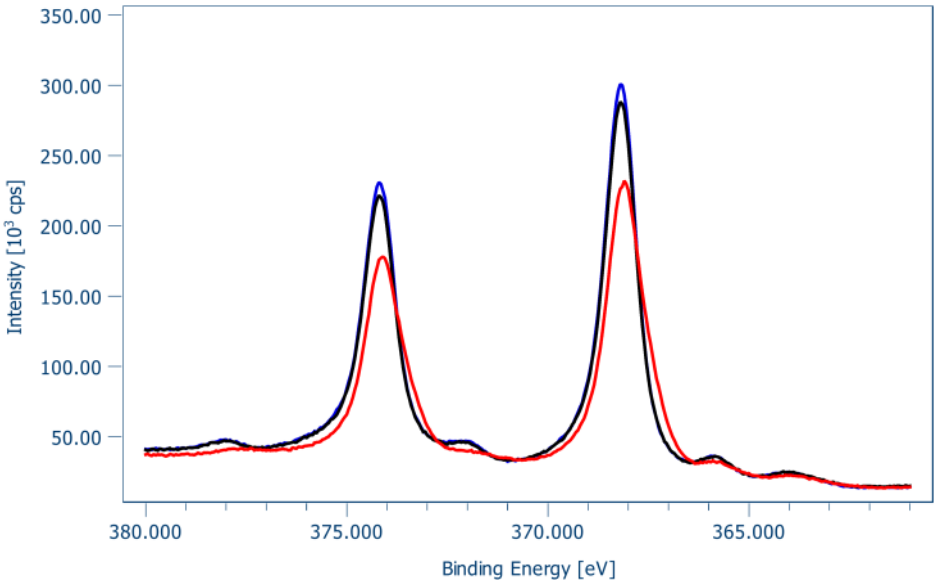
Temperature profile recorded with QMS: *200203-file2* (T at beginning 286 K)

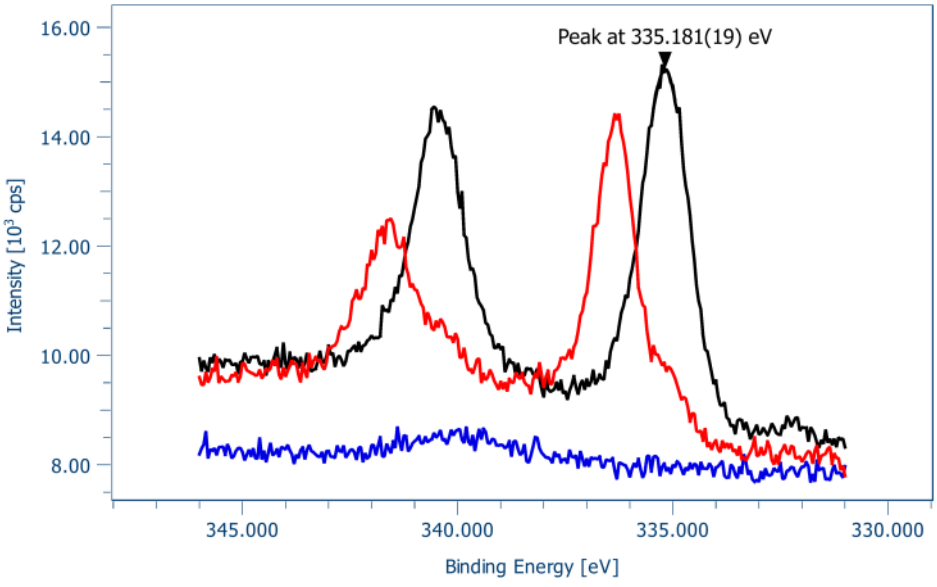


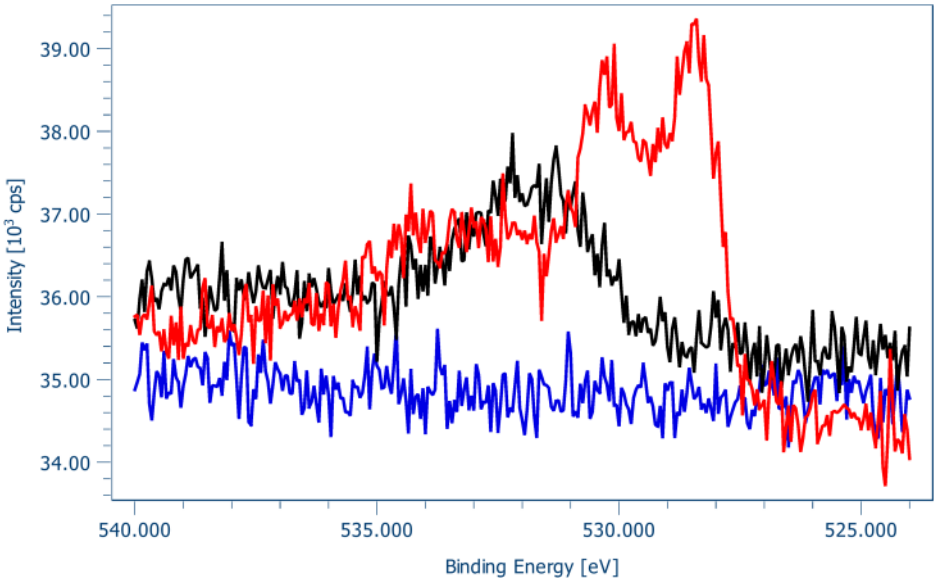
Switch back to air cooling for H2 dose; clear water from cryostat
Shut valve to HP cell at 15.0"
Turn on X-ray source at 15.0"

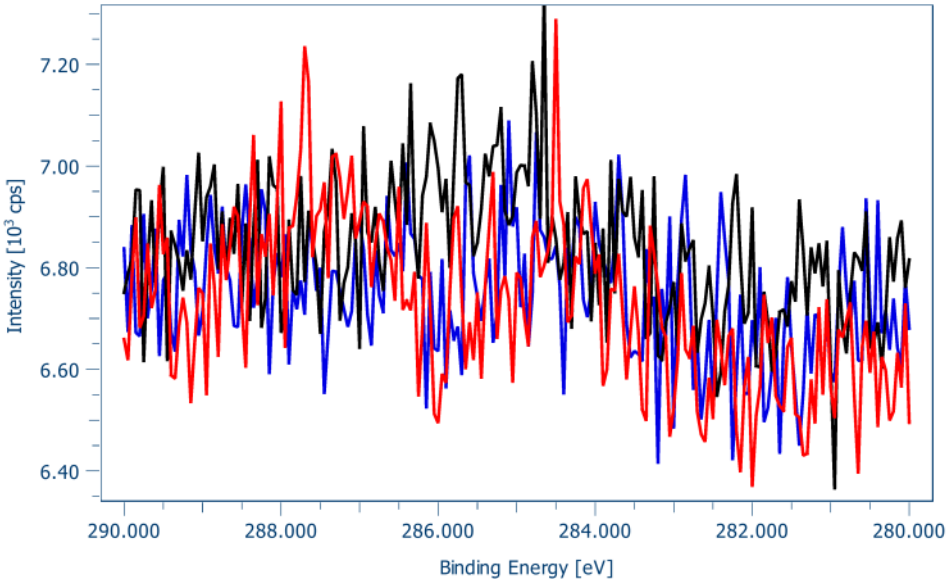
O2 pre-stage pressure was 1925 psi at 11:15 PM

Measure
XPS: after oxidation#2 - 66 degrees



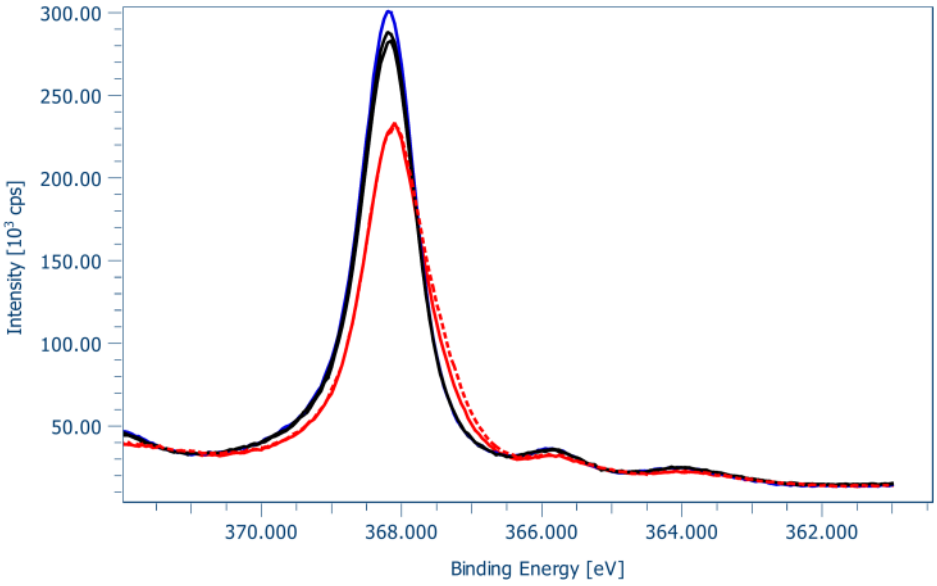


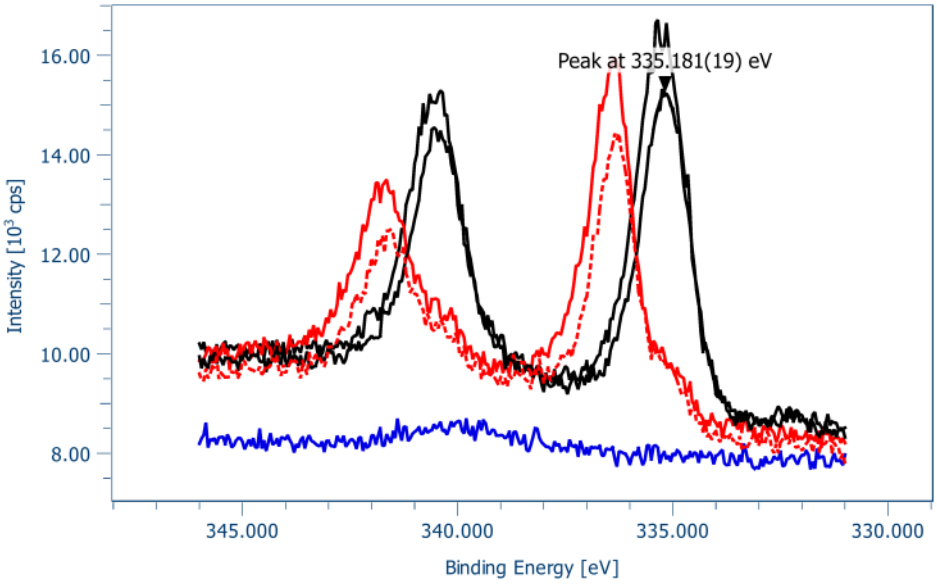


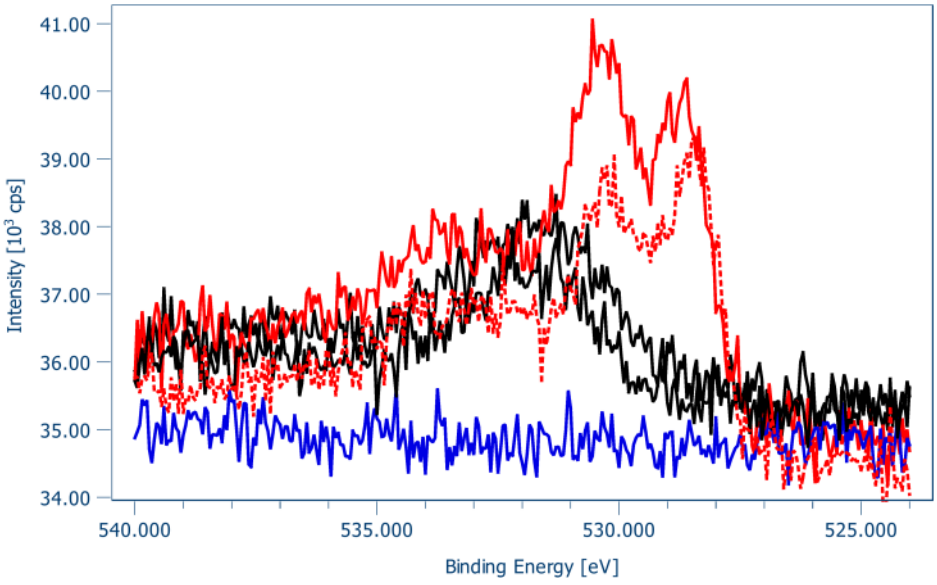


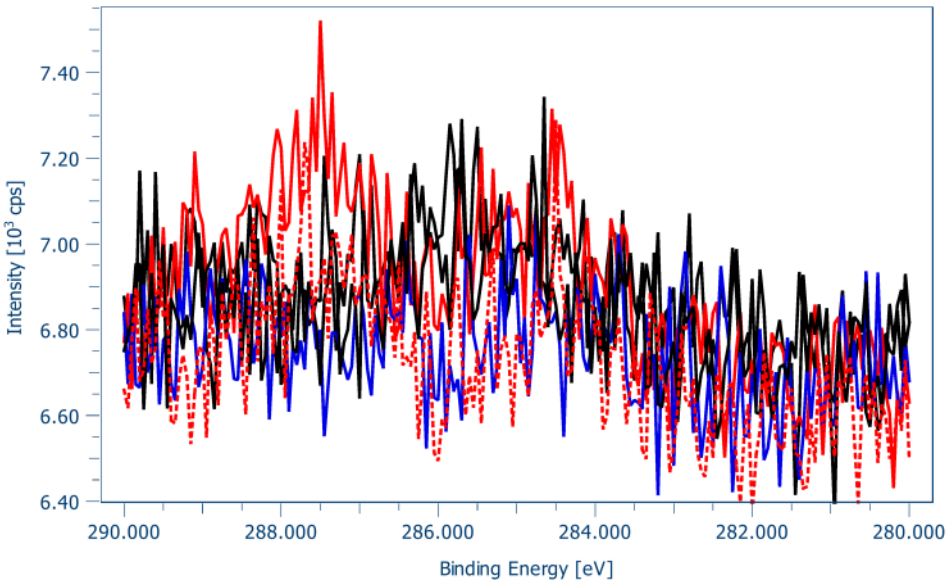
Comparison to first oxidation today: 2nd round in dashed lines

Calling this reproducible


300.00
250.00
200.00
150.00
100.00
50.00
Intensity [10^3 cps]
370.000
368.000
366.000
364.000
362.000
Binding Energy [eV]

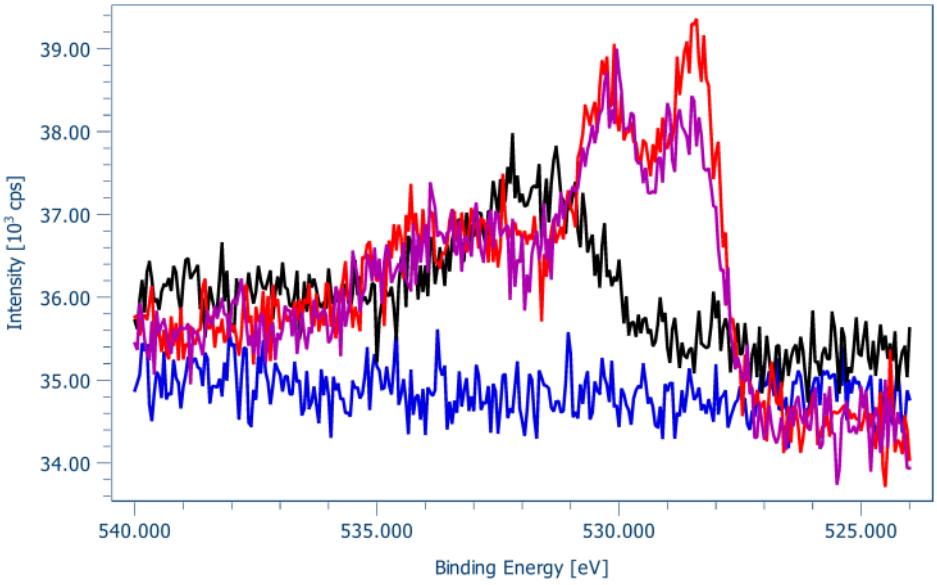

Peak at 335.181(19) eV
16.00
14.00
12.00
10.00
8.00
Intensity [10^3 cps]
345.000
340.000
335.000
330.000
Binding Energy [eV]

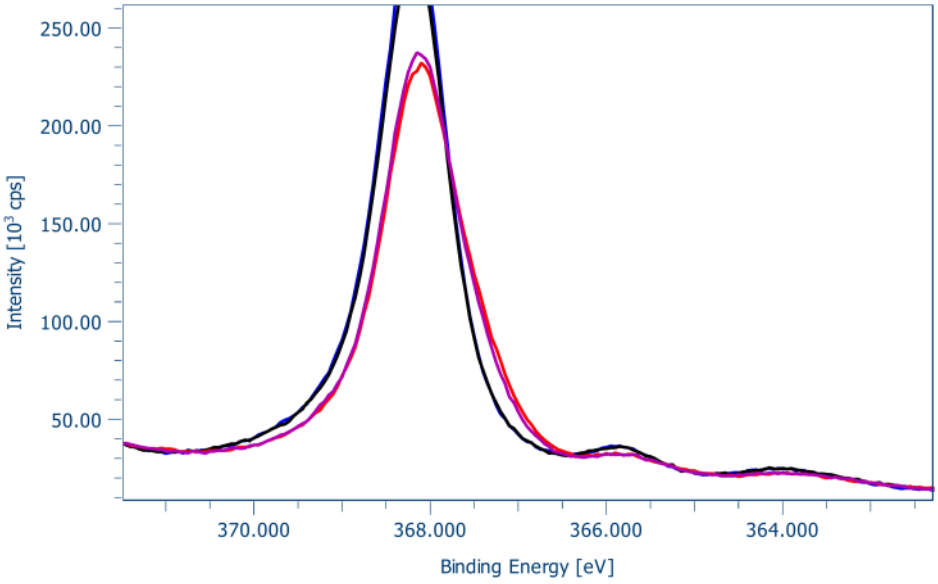

41.00
40.00
39.00
38.00
37.00
36.00
35.00
34.00
Intensity [10^3 cps]
540.000
535.000
530.000
525.000
Binding Energy [eV]

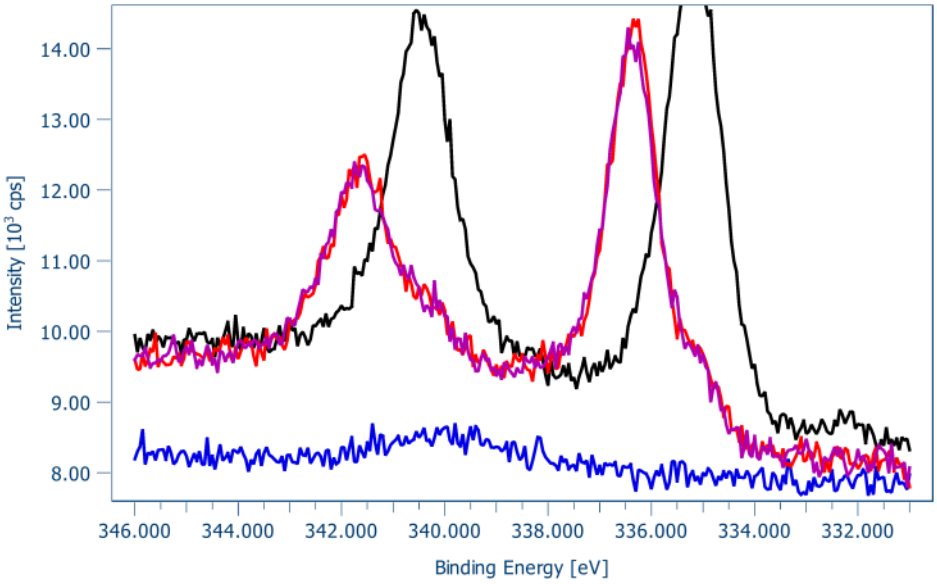


30 mins exposure to background with X-ray gun off

XPS: 30 mins bkg#2 - 66 degrees
purple







Start sputter 5:00 PM

- Sputter for 30 mins at 20 mA and 3e-5 Torr Ar
- Anneal to 800 K for 10 min

Conclusion:
State of chamber not changing
Established protocol which has to be followed! (#EXPTL SUM / 200203 - O2 Protocol.docx)
0.09 or 0.14 ML doesn't make a difference as expected

200204 - PdAg + 575 & 600 K O2 + H2

Tuesday, February 4, 2020 9:53 AM

Plan

Oxidation temp where PdO 100% oxidized

Logbook

Legend - Plan in purple

Flash anneal to 800 K
Set up water cooling
Open gate valve

Measure clean Ag and turn down O3 and off (8:50 AM)
XPS: clean Ag - 66 degrees
Switch on evap

- **Pd evaporation**: 2.50 A, 1000 V, 9.7 mA, 1.1 nA for **2 min 00 sec** at 300K
  Goal: 0.12 ML
  0.12/0.0533=2.2514

Connect cooling
9:55 AM

Switch on X-Ray source before transfer and turn ion gauge off

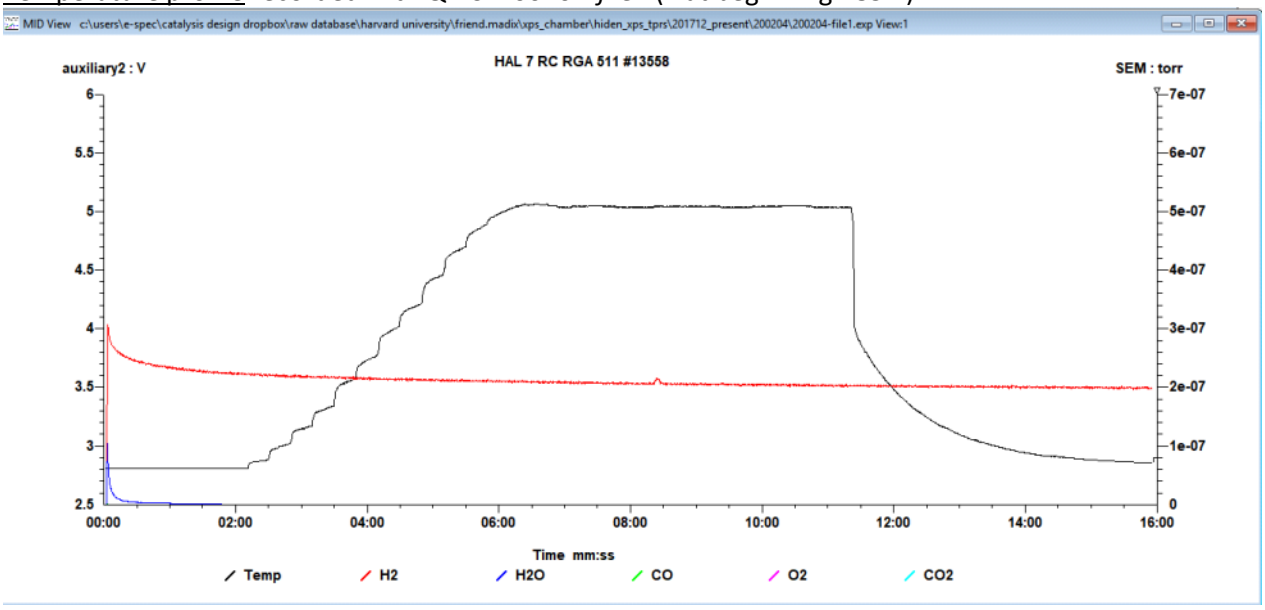
XPS: Pd deposition - 0 degrees
- Pd area: 15 kcps eV
- Ag area: 702 kcps eV
- Pd/Ag: 15/702 = 0.0214 --> **0.12(8) ML GREAT**

XPS: Pd deposition - 66 degrees
- Pd area: 14 kcps eV
- Ag area: 268 kcps eV
- Pd/Ag: 14/268 = 0.0522 --> **0.14(0) ML TOP**

Done at 10:30 AM

Turn up O2 at 10:45 AM
- **Oxidized** in 1.93(2) Torr O2 at 575 K for **5 min**
  - Valve cracked
  - O2 flowing from O3 conditions before
  - turbo 19 W
  - 1, 3, 5, 7, 10, 12, 15, 17, 20, 23, 25 W to ramp (in 20 sec intervals); let climb to 575 K at 25 W (started timer when it hit 575 K); 23.5 W laser power to stabilize at 550 K
  - maintained T within 575-579 K over duration of oxidation
  - Cooling in O2 till T = 305 K (~1 min) , then evacuating HP cell till p_baratron = 0.1 mTorr 11:12 AM

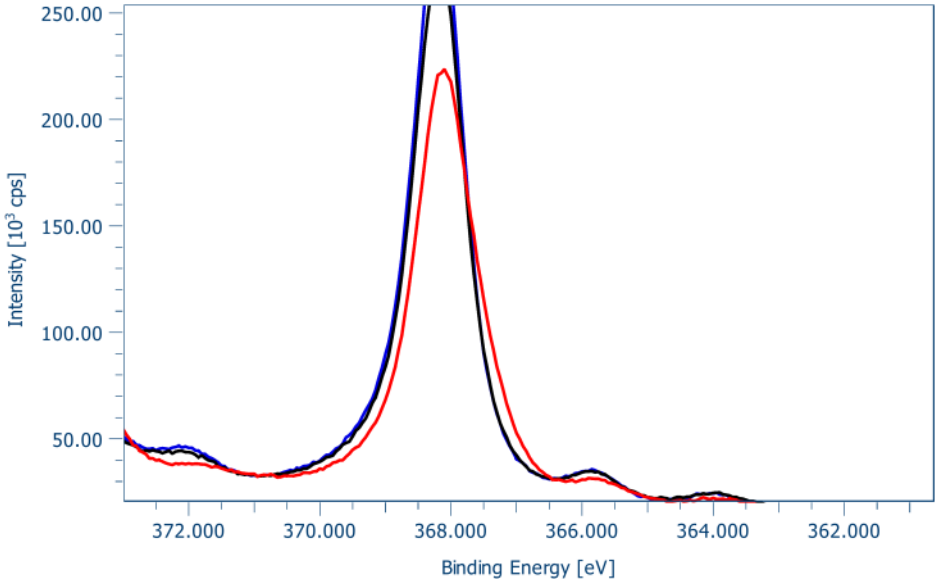
Temperature profile recorded with QMS: *200204-file1* (T at beginning 285 K)

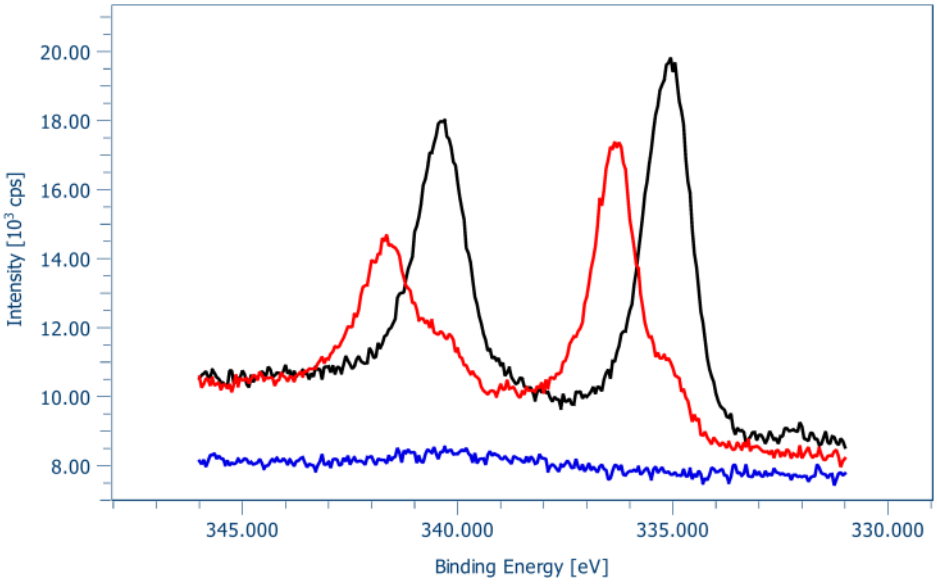


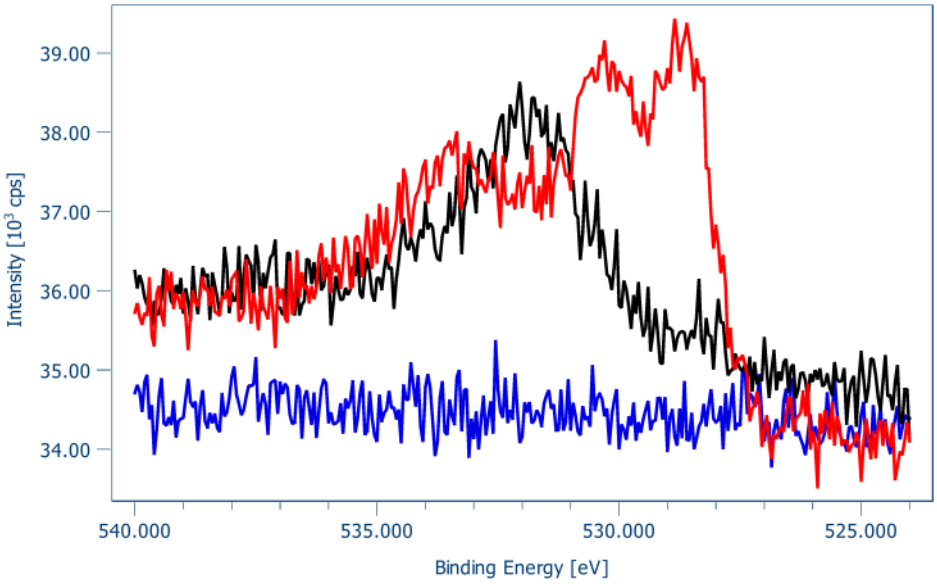
Switch back to air cooling for H2 dose; clear water from cryostat
Shut valve to HP cell at 15.0"
Turn on X-ray source at 15.0"

O2 pre-stage pressure was 1575 psi at 11:12 AM

Measure (11:22 AM)
XPS: after oxidation - 66 degrees
Ag seems a bit less oxidized than 550 K
Pd oxidation pretty similar to 550 K







XPS: after oxidation - 0 degrees

Pd buried in deeper layers?

Done at 11:44 AM

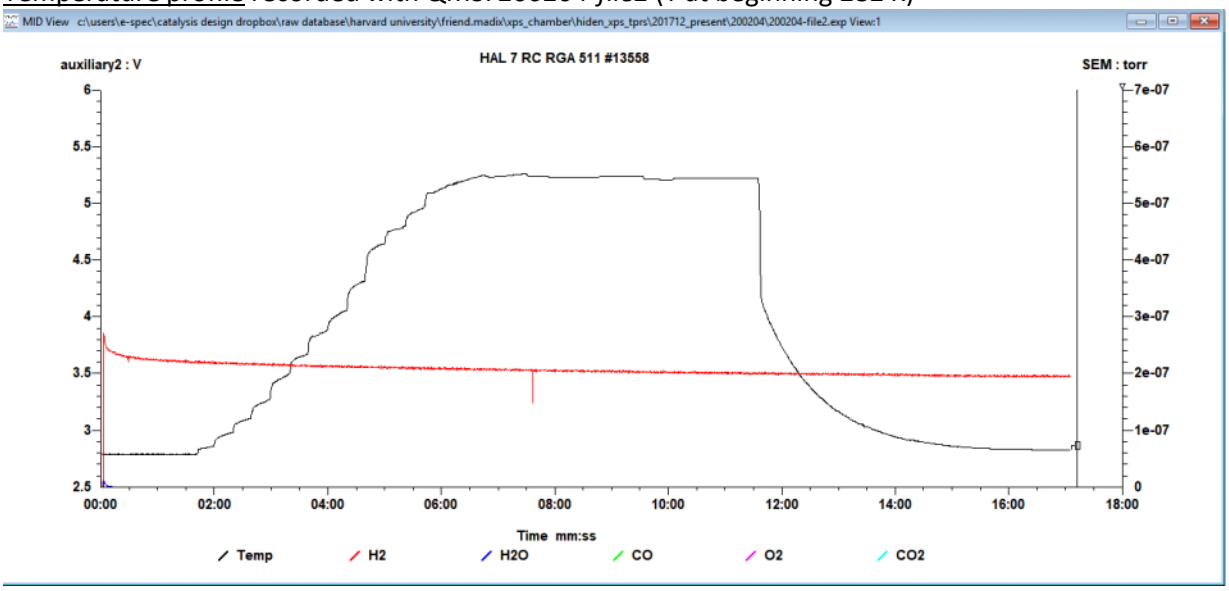
####################
2nd oxidation (subsequent)
####################

Turn up O2 at 12:20 PM

- **Oxidized** in 1.93(7) Torr O2 at 600 K for **5 min**
  - Valve cracked
  - O2 flowing from O3 conditions before
  - turbo 19 W
  - 1, 3, 5, 7, 10, 12, 15, 17, 20, 23, 25, 27, 29 W to ramp (in 20 sec intervals); let climb to 600 K at 29 W (started timer when it hit 600 K); 27.8 W laser power to stabilize at 600 K
  - maintained T within 599-604 K over duration of oxidation

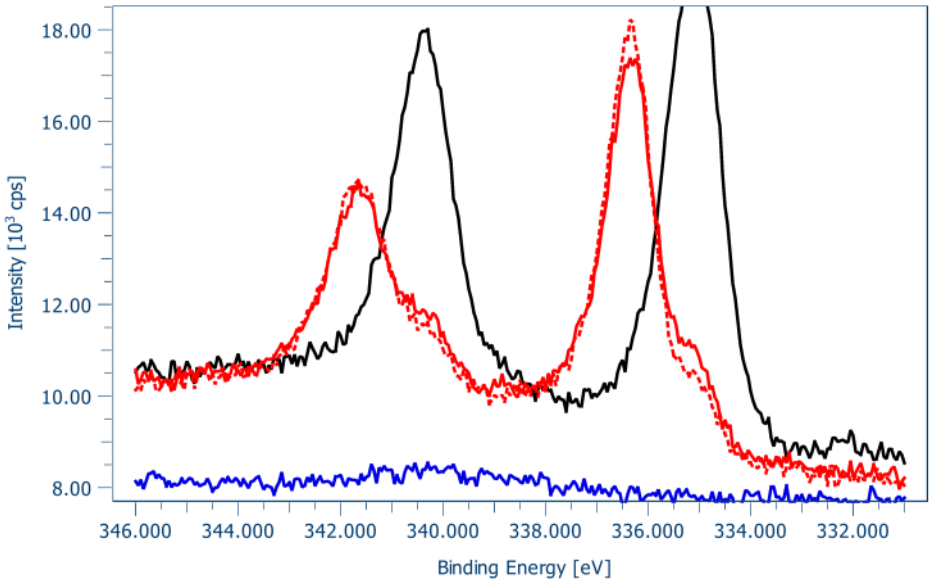
- Cooling in O2 till T = 305 K (~1 min) , then evacuating HP cell till p_baratron = 0.1 mTorr 12:40 PM
- H2O cooling was very efficient this time

Temperature profile recorded with QMS: *200204-file2* (T at beginning 282 K)



Switch back to air cooling for H2 dose; clear water from cryostat
Shut valve to HP cell at 15.0"
Turn on X-ray source at 15.0"

Measure (12:50 PM)
XPS: after oxidation - 66 degrees
Almost equal compared to previous 550 K oxidation



- **H2 Dose#1**: 2e-4 H2 for 5 min at 300 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  XPS: H2 Dose#1 - 66 degrees

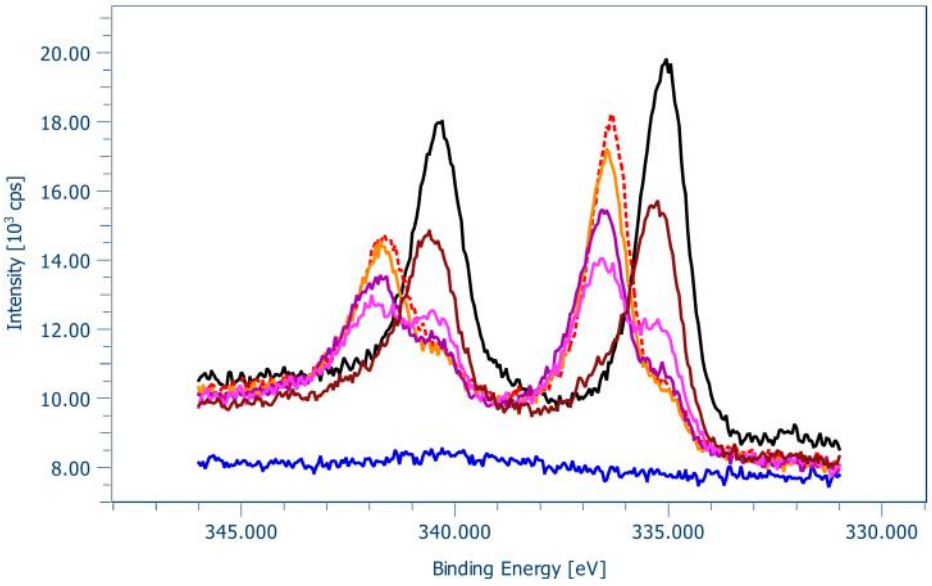
- **H2 Dose#2**: 2e-4 H2 for 15 min (<u>total: 20 min</u>) at 300 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)

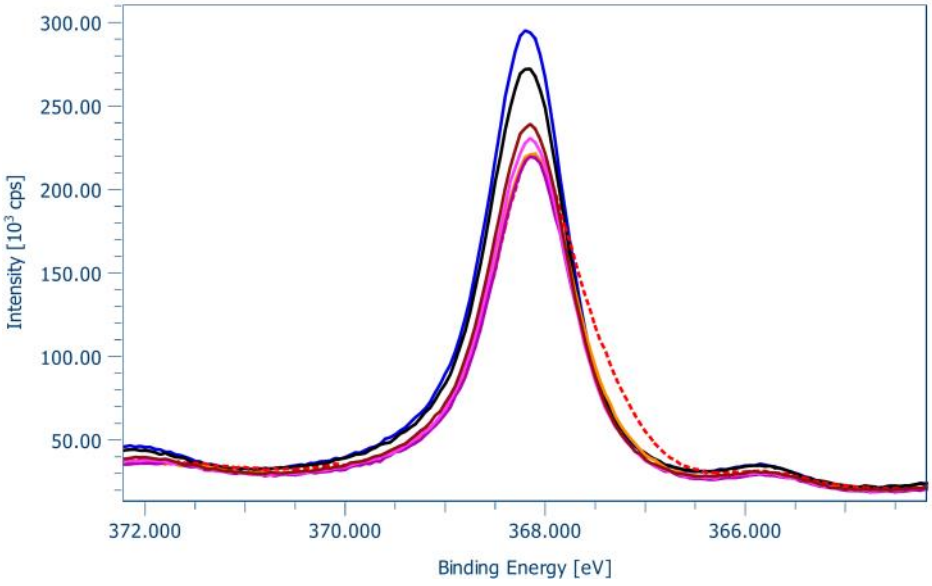
XPS: H2 Dose#2 - 66 degrees

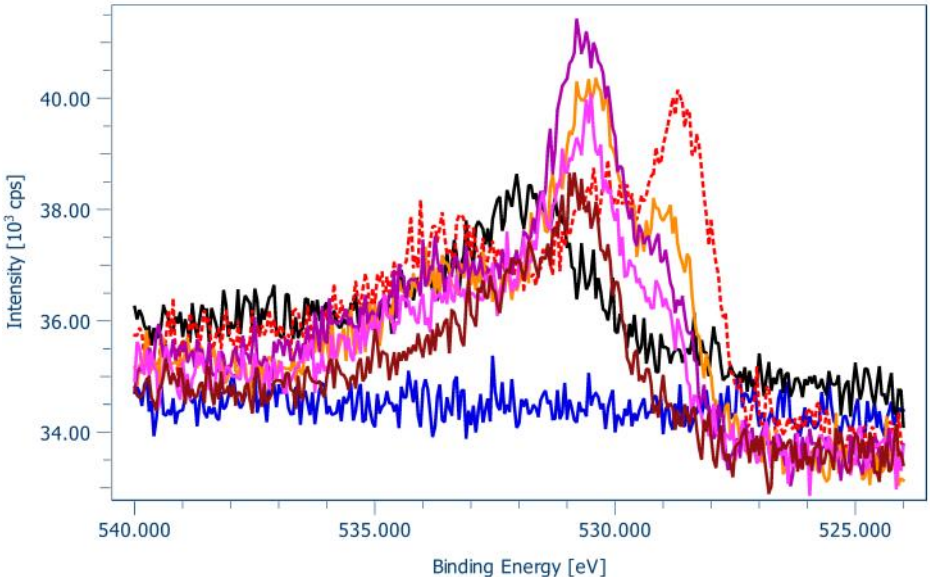
- **H2 Dose#3**: 2e-4 H2 for 15 min (<u>total: 35 min</u>) at 300 K (background, right leak valve) (1.0e-4 in prep; had analysis ion gauge off)
  XPS: H2 Dose#3 - 66 degrees

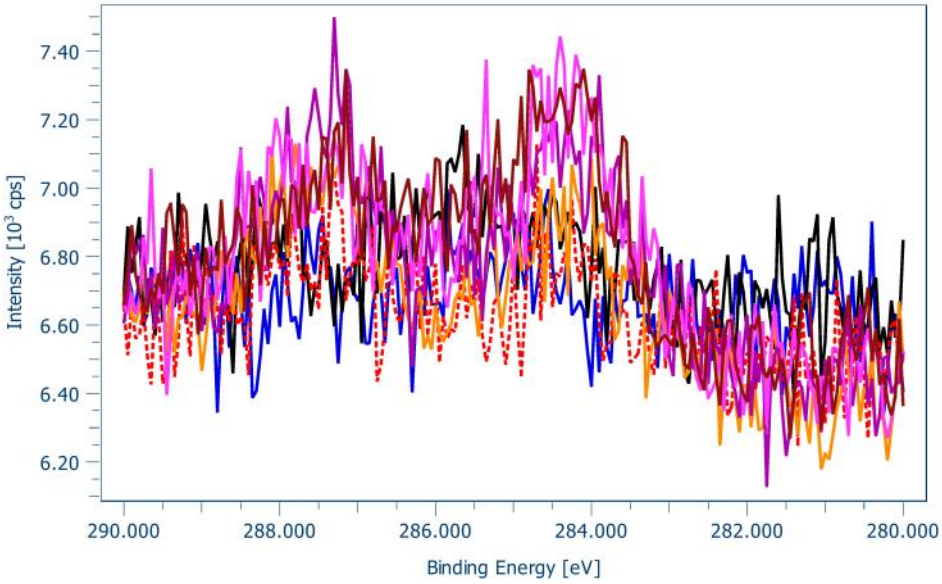
- **H2 Dose#4**: 2e-4 H2 for 15 min (<u>total: 50 min</u>) at 300 K (background, right leak valve) (1.0e-4 in prep; had analysis ion gauge off)
  XPS: H2 Dose#4 - 66 degrees

Pd deposition
After oxidation#2
H2 Dose#1
H2 Dose#2
H2 Dose#3
H2 Dose#4









Conclusion

1. **Differences of higher (600 K) and lower (550 K) T oxidation are small; choose rather 550 K since this is closer to conditions making p4x4**
2. **Time steps for Pd at E-4 Torr H2: 5, 20, 35, 40, 45, 50 + min**
3. **Additional time steps for Ag at E-4 Torr H2: 1, 3 min**
4. **Do also 1 min at E-7 Torr H2**

200206 - PdAg + 550 K O2 + H2 [1st cycle]

Thursday, February 6, 2020 9:01 AM

Flash anneal to 800 K
Set up water cooling
Open gate valve

Turn down O3 and off (9:00 AM)

Measure clean Ag
XPS: clean Ag - 66 degrees

Connect cooling
10:00 AM

- **Pd evaporation**: 2.50 A, 1000 V, 9.6 mA, 1.2 - 0.6 nA for **2 min 00 sec** at 300K
  Goal: 0.12 ML

Switch on X-Ray source before transfer and turn ion gauge off

XPS: Pd deposition - 0 degrees
  - Pd area: 8.3 kcps eV
  - Ag area: 686 kcps eV
  - Pd/Ag: 8.3/686 = 0.0121 --> **0.07(3) ML Not enough**

- **Pd evaporation#2**: 2.50 A, 1000 V, 9.6 mA, 0.7 nA for **1 min 30 sec** at 300K
  Goal: 0.12 ML
  0.07/2=0.035 ML/min
  0.12-0.07 = 0.05 ML
  0.05/0.035=1.4286 min
  0.43*60=25.8 sec

XPS: Pd deposition#2 - 0 degrees
  - Pd area: 14 kcps eV
  - Ag area: 686 kcps eV
  - Pd/Ag: 14/686 = 0.0204 --> **0.12(2) ML GREAT**

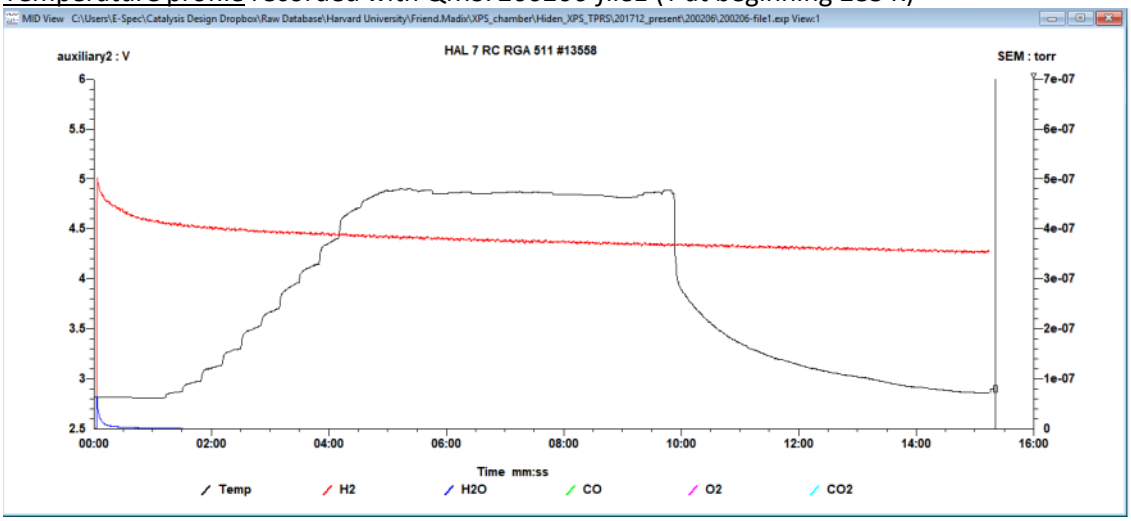
Start at 10:34 AM
XPS: Pd deposit0ion#2 - 66 degrees
  - Pd area: 12.6 kcps eV
  - Ag area: 262 kcps eV
  - Pd/Ag: 12.6/262 = 0.0481 --> **0.12(9) ML NICE**

Done at 10:54 AM

Turn up O2 at 11:04 AM
- **Oxidized** in 1.93(1) Torr O2 at 550 K for **5 min**
  - Valve cracked
  - O2 flowing from O3 conditions before
  - turbo 19 W

- 1, 3, 5, 7, 10, 12, 15, 17, 20, 23, 24 W to ramp (in 20 sec intervals); let climb to 575 K at 25 W (started timer when it hit 550 K); 20.5 W laser power to stabilize at 550 K
- maintained T within 550-558 K over duration of oxidation
- Cooling in O2 till T = 305 K (~1 min) , then evacuating HP cell till p_baratron = 0.1 mTorr 11:36 AM

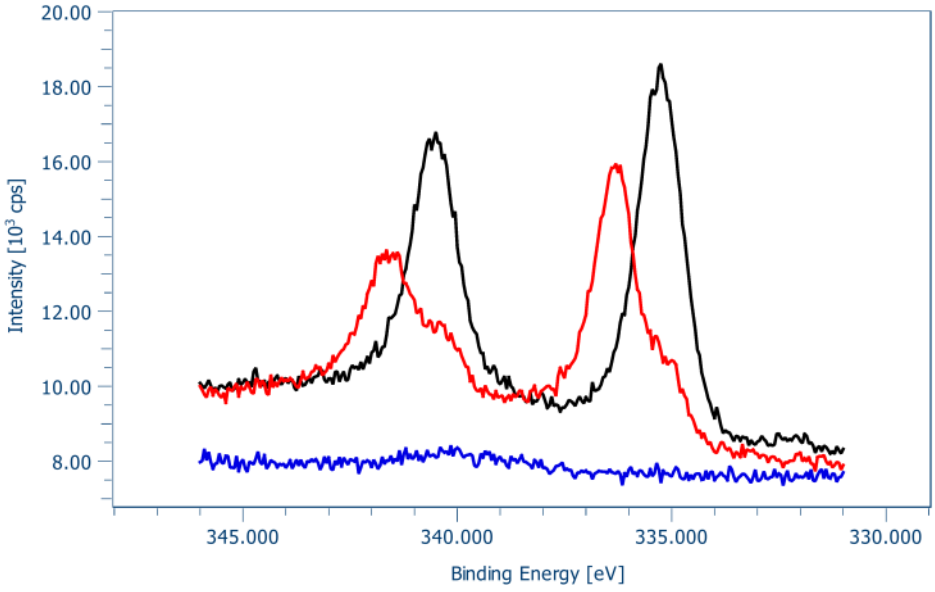
Temperature profile recorded with QMS: *200206-file1* (T at beginning 285 K)



Switch back to air cooling for H2 dose; clear water from cryostat
Shut valve to HP cell at 15.0"
Turn on X-ray source at 15.0"

O2 pre-stage pressure was 825 psi at 11:36 AM

Measure (11:47 AM)
XPS: after oxidation - 66 degrees



Closed O2 valves, stop O2 flow behind HP O2 leak valve

- **H2 Dose#1**: 1e-7 H2 for 1 min at 295 K (background, right leak valve)
  (5e-8 in prep; had analysis ion gauge off)
  XPS: H2 Dose#1 - 66 degrees

p_prep = 3.7e-9
Done 12:09 PM

- **H2 Dose#2**: 1e-5 H2 for 1 min at 296 K (background, right leak valve)
  (5.0e-6 in prep; had analysis ion gauge off)
  XPS: H2 Dose#2 - 66 degrees
  p_prep = 6e-9 - 4.7e-9
  Done 12:22 PM

- **H2 Dose#3**: 1e-4 H2 for 1 min at 297 K (background, right leak valve)
  (5.0e-5 in prep; had analysis ion gauge off)
  Wait 2.5 min to recover pressure
  XPS: H2 Dose#3 - 66 degrees
  p_prep =1.9e-8
  Done 12:39 PM

- **H2 Dose#4**: 2e-4 H2 for 1 min (total: 1 min) at 298 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 2.5 min to recover pressure
  XPS: H2 Dose#4 - 66 degrees
  p_prep = 2.6e-8
  Done 12:53 PM

- **H2 Dose#5**: 2e-4 H2 for 1 min (total: 2 min) at 300 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 2.5 min to recover pressure
  XPS: H2 Dose#5 - 66 degrees
  p_prep = 2.0e-8
  Done 1:11 PM

- **H2 Dose#6**: 2e-4 H2 for 3 min (total: 5 min) at 300 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#6 - 66 degrees
  p_prep = 2.5e-8
  Done 1:29 PM

- **H2 Dose#7**: 2e-4 H2 for 3 min (total: 8 min) at 300 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#7 - 66 degrees
  p_prep = 2.1e-8
  Done at 1:49 PM
  Regime of autocatalytic reduction of PdO

- **H2 Dose#8**: 2e-4 H2 for 1 min (total: 9 min) at 300 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#8 - 66 degrees
  p_prep = 2.1e-8
  Done at 2:08 PM
  Regime of autocatalytic reduction of PdO

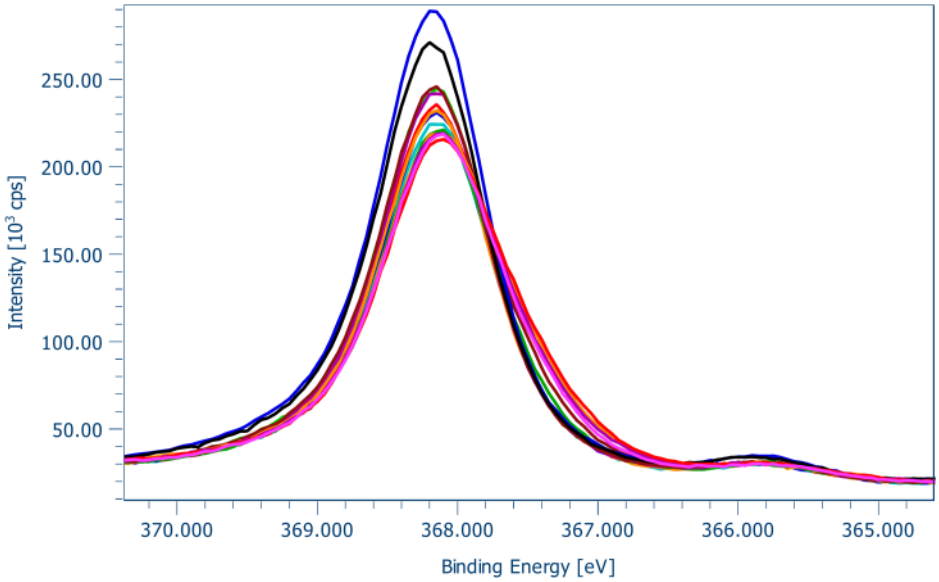
- **H2 Dose#9**: 2e-4 H2 for 1 min (total: 10 min) at 300 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#9 - 66 degrees
    p_prep = 2.2e-8
    Done at 2:25 PM
    Regime of autocatalytic reduction of PdO

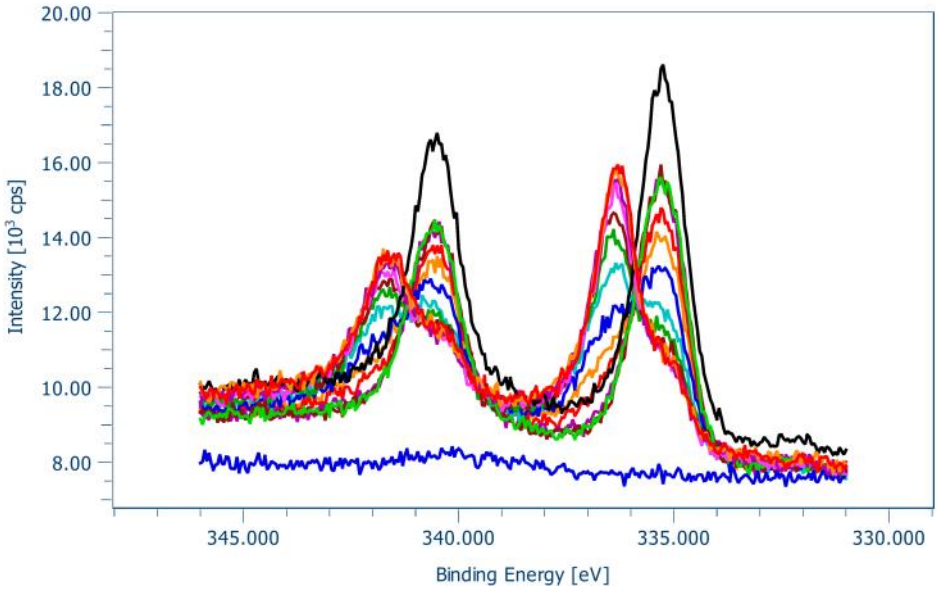
- **H2 Dose#10**: 2e-4 H2 for 5 min (total: 15 min) at 300 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#10 - 66 degrees
    p_prep = 2.3e-8
    Done at 2:48 PM

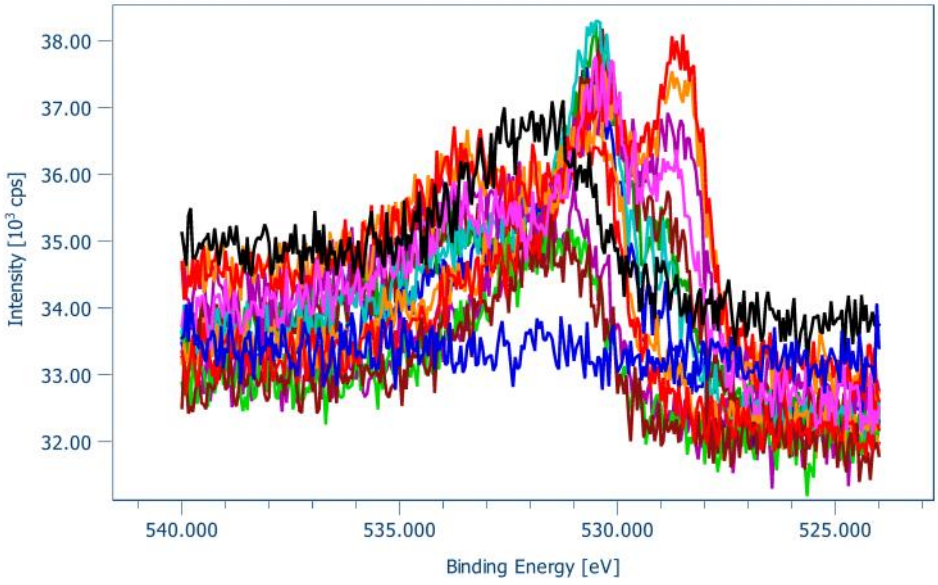
- **H2 Dose#11**: 2e-4 H2 for 5 min (total: 20 min) at 300 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#11 - 66 degrees
    p_prep = 2.3e-8
    Done at 3:11 PM
    Seems like reduction is complete, but doing one more spectra to be sure

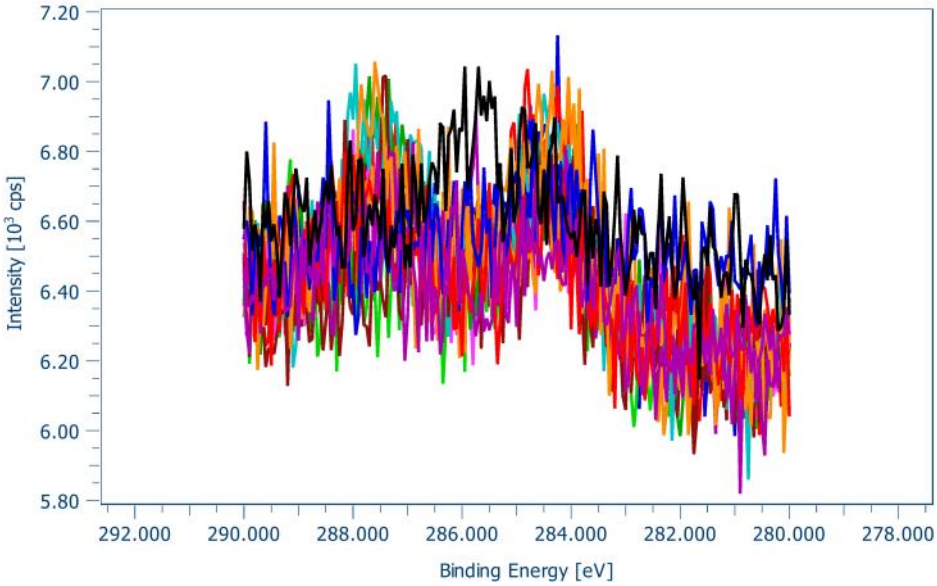
- **H2 Dose#12**: 2e-4 H2 for 10 min (total: 30 min) at 300 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#12 - 66 degrees
    p_prep = 2.3e-8
    Done at 3:40 PM
    Overlapping with last spectra (#11) --> DONE

Pd deposition#2
After oxidation
H2 Dose#1
H2 Dose#2
H2 Dose#3
H2 Dose#4
H2 Dose#5
H2 Dose#6
H2 Dose#7
H2 Dose#8
H2 Dose#9
H2 Dose#10
H2 Dose#11
H2 Dose#12


250.00
200.00
150.00
100.00
50.00
Intensity [10³ cps]
370.000
369.000
368.000
367.000
366.000
365.000
Binding Energy [eV]


20.00
18.00
16.00
14.00
12.00
10.00
8.00
Intensity [10³ cps]
345.000
340.000
335.000
330.000
Binding Energy [eV]


38.00
37.00
36.00
35.00
34.00
33.00
32.00
Intensity [10³ cps]
540.000
535.000
530.000
525.000
Binding Energy [eV]



Start O2 flow at 6 PM
Turn up ozone at 6:20 PM

200207 - PdAg + 550 K O2 + H2 [2nd cycle]

Friday, February 7, 2020 8:52 AM

Turn down O3 and off (8:45 AM)

Measure
XPS: Ag after 1st reduction - 66 degrees

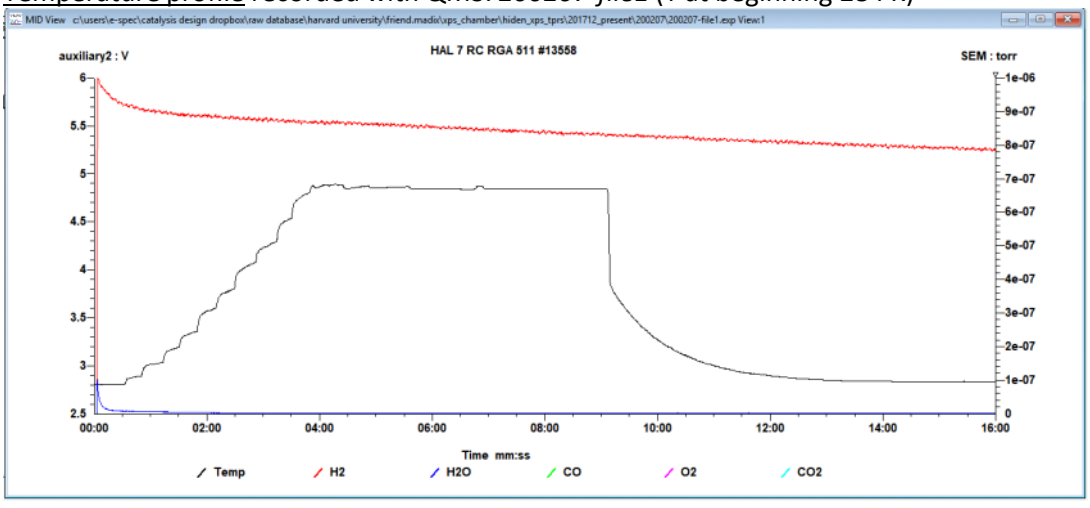
Need to leave 1 h 30 mins in UHV (at least till 10:15 AM) between turning down O3 and oxidizing

Connect cooling
9:10 AM

Turn up O2 at 10:30 AM

- **Oxidized** in 1.92(9) Torr O2 at 550 K for **5 min**
  - Valve cracked
  - O2 flowing from O3 conditions before
  - turbo 19 W
  - 1, 3, 5, 7, 10, 12, 15, 17, 20, 23, 24 W to ramp (in 20 sec intervals); let climb to 550 K at 24 W (started timer when it hit 550 K); 21.5 W laser power to stabilize at 550 K
  - maintained T within 550-556 K over duration of oxidation
  - Cooling in O2 till T = 305 K (~1 min) , then evacuating HP cell till p_baratron = 0.1 mTorr 11:00 AM

Temperature profile recorded with QMS: *200207-file1* (T at beginning 284 K)



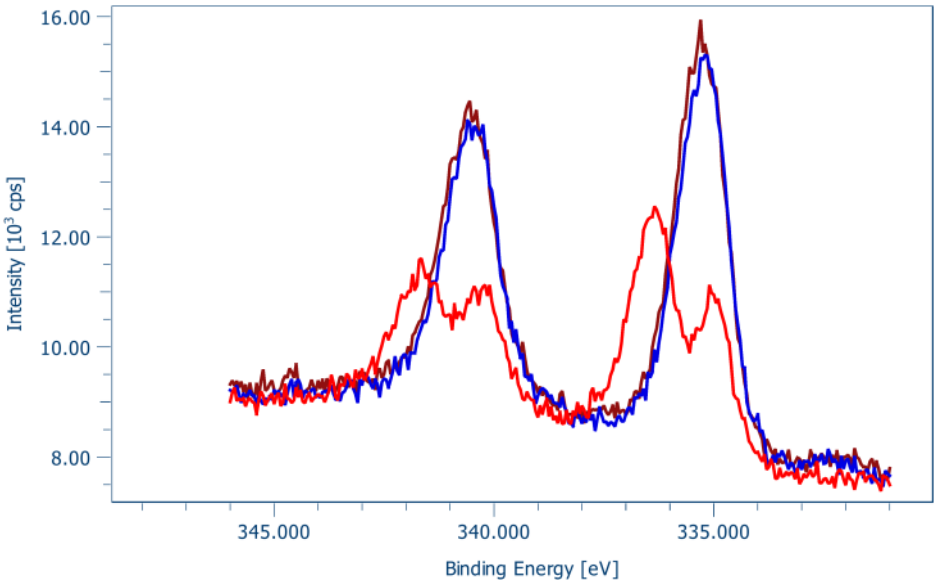
Switch back to air cooling for H2 dose; clear water from cryostat
Shut valve to HP cell at 15.0"
Turn on X-ray source at 15.0"

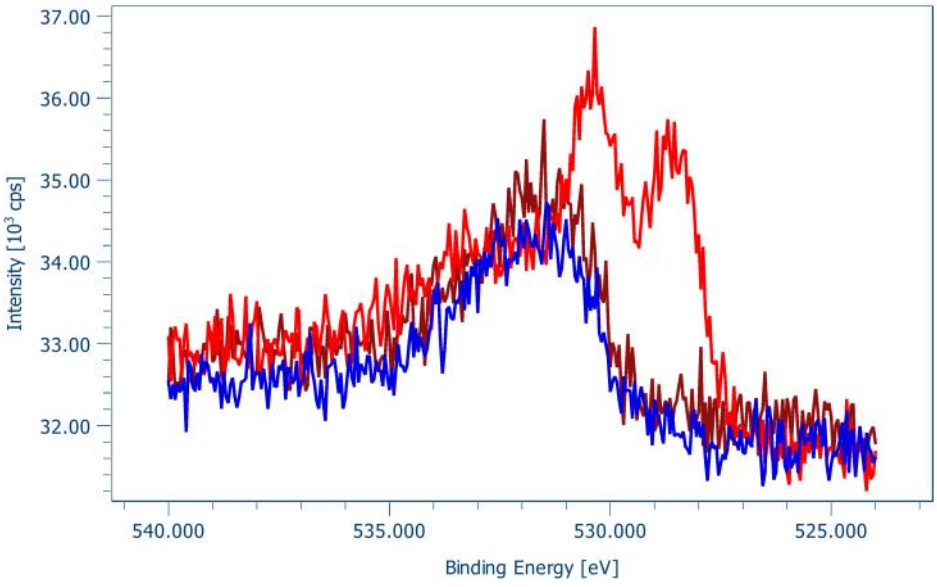
O2 pre-stage pressure was 550 psi at 11:15 AM

Measure (11:11 AM)
XPS: after oxidation - 66 degrees

H2 Dose#12

Ag after 1st cycle
After oxidation





SAME DOSES AS 1ST CYCLE

- **H2 Dose#1**: 1e-7 H2 for 1 min at 294 K (background, right leak valve)
  (5.0e-8 in prep; p_bkg = 6e-9; p_delta = 4.4e-8; had analysis ion gauge off)
  XPS: H2 Dose#1 - 66 degrees
  p_prep = 6.3e-9
  Done 11:36 AM

- **H2 Dose#2**: 1e-5 H2 for 1 min at 295 K (background, right leak valve)
  (5.0e-6 in prep; had analysis ion gauge off)
  XPS: H2 Dose#2 - 66 degrees
  p_prep = 8e-9
  Done 11: 48 AM

- **H2 Dose#3**: 1e-4 H2 for 1 min at 296 K (background, right leak valve)

(5.0e-5 in prep; had analysis ion gauge off)
Wait 2.5 min to recover pressure
XPS: H2 Dose#3 - 66 degrees
p_prep = 2.0e-8
Done 12:06 PM

- **H2 Dose#4**: 2e-4 H2 for 1 min (total: 1 min) at 296 K (background, right leak valve)
(1.0e-4 in prep; had analysis ion gauge off)
Wait 2.5 min to recover pressure
XPS: H2 Dose#4 - 66 degrees
p_prep = 2.3e-8
Done 12:22 PM

- **H2 Dose#5**: 2e-4 H2 for 1 min (total: 2 min) at 296 K (background, right leak valve)
(1.0e-4 in prep; had analysis ion gauge off)
Wait 2.5 min to recover pressure
XPS: H2 Dose#5 - 66 degrees
p_prep = 2.3e-8
Done 12:38 PM

- **H2 Dose#6**: 2e-4 H2 for 3 min (total: 5 min) at 297 K (background, right leak valve)
(1.0e-4 in prep; had analysis ion gauge off)
Wait 3 min to recover pressure
XPS: H2 Dose#6 - 66 degrees
p_prep = 2.2e-8
Done 12:59 PM

- **H2 Dose#7**: 2e-4 H2 for 3 min (total: 8 min) at 297 K (background, right leak valve)
(1.0e-4 in prep; had analysis ion gauge off)
Wait 3 min to recover pressure
XPS: H2 Dose#7 - 66 degrees
p_prep = 2.3e-8
Done at 1:19 PM
Regime of autocatalytic reduction of PdO

- **H2 Dose#8**: 2e-4 H2 for 1 min (total: 9 min) at 297 K (background, right leak valve)
(1.0e-4 in prep; had analysis ion gauge off)
Wait 3 min to recover pressure
XPS: H2 Dose#8 - 66 degrees
p_prep = 2.1e-8
Done at 1:38 PM
Regime of autocatalytic reduction of PdO

- **H2 Dose#9**: 2e-4 H2 for 1 min (total: 10 min) at 297 K (background, right leak valve)
(1.0e-4 in prep; had analysis ion gauge off)
Wait 3 min to recover pressure
XPS: H2 Dose#9 - 66 degrees
p_prep = 2.2e-8
Done at 1:55 PM
Regime of autocatalytic reduction of PdO

- **H2 Dose#10**: 2e-4 H2 for 5 min (total: 15 min) at 297 K (background, right leak valve)
(1.0e-4 in prep; had analysis ion gauge off)

Wait 3 min to recover pressure
XPS: H2 Dose#10 - 66 degrees
p_prep = 2.1e-8
Done at 2:19 PM

- **H2 Dose#11**: 2e-4 H2 for 5 min (total: 20 min) at 297 K (background, right leak valve)
(1.0e-4 in prep; had analysis ion gauge off)
Wait 3 min to recover pressure
XPS: H2 Dose#11 - 66 degrees
p_prep = 2.5e-8
Done at 2:40 PM

- **H2 Dose#12**: 2e-4 H2 for 10 min (total: 30 min) at 297 K (background, right leak valve)
(1.0e-4 in prep; had analysis ion gauge off)
Wait 3 min to recover pressure
XPS: H2 Dose#12 - 66 degrees
p_prep = 2.5e-8
Done at 3:09 PM

- **H2 Dose#13**: 2e-4 H2 for 10 min (total: 40 min) at 297 K (background, right leak valve)
(1.0e-4 in prep; had analysis ion gauge off)
Overshot to 2.2e-4 in prep
Wait 3 min to recover pressure
XPS: H2 Dose#13 - 66 degrees
p_prep = 2.6e-8
Done at 3:40 PM
Spectra overlapping with #12, but small bump in O1s --> Do one last dose to make sure

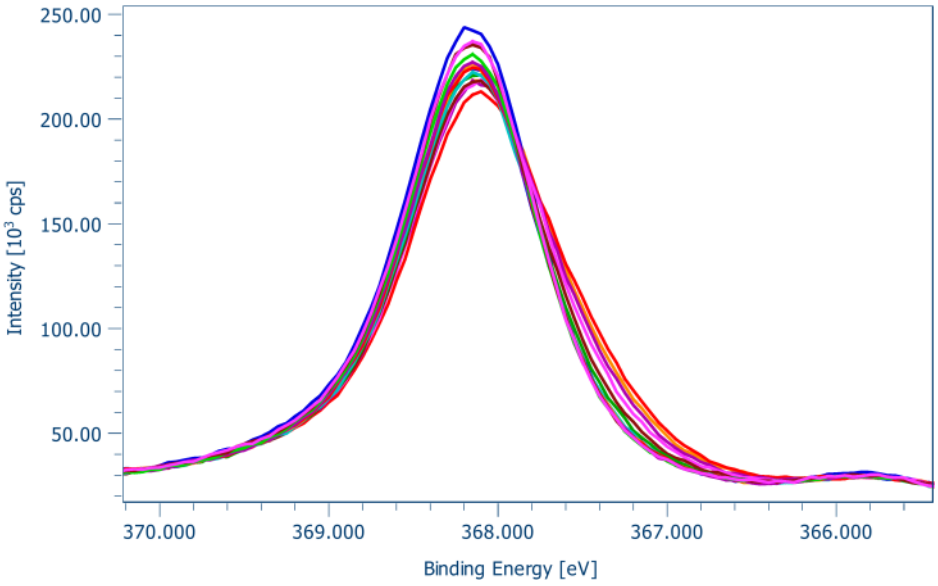
- **H2 Dose#14**: 2e-4 H2 for 10 min (total: 50 min) at 297 K (background, right leak valve)
(1.0e-4 in prep; had analysis ion gauge off)
Wait 3 min to recover pressure
XPS: H2 Dose#14 - 66 degrees
p_prep = 2.e-8
Done at 4:13 PM
Overlapping --> DONE!

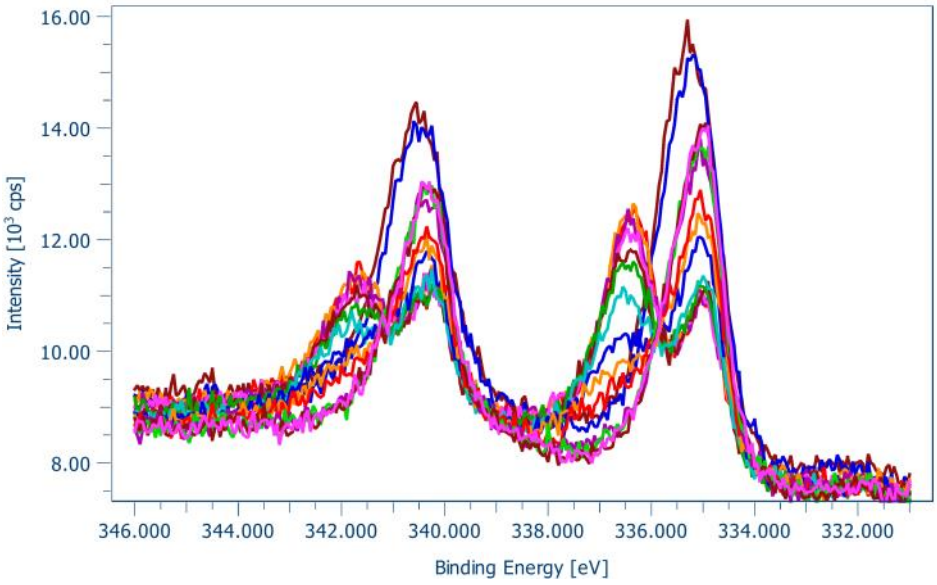
Don't forget VB spectrum!!

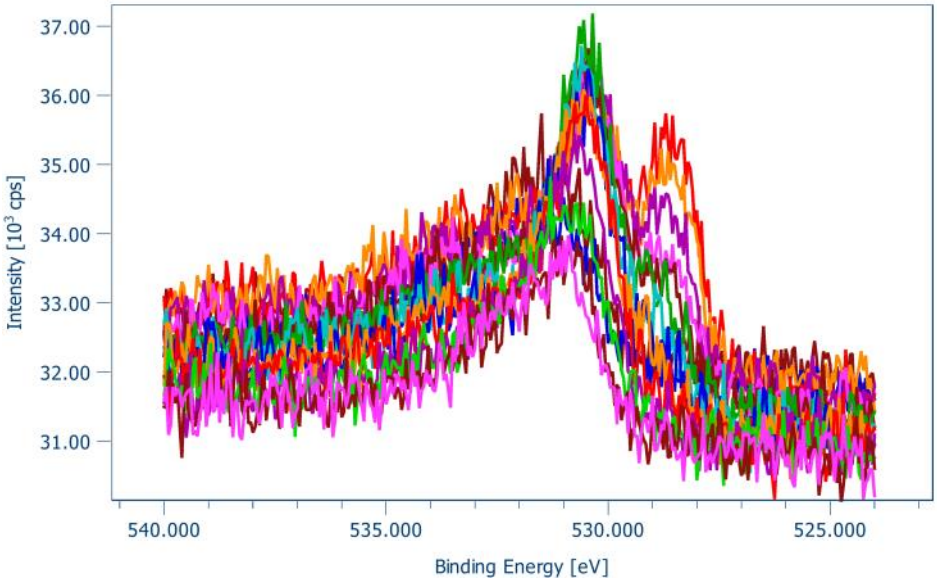
Legend:
H2 Dose#12 (day before)
After oxidation
H2 Dose#1
H2 Dose#2
H2 Dose#3
H2 Dose#4
H2 Dose#5
H2 Dose#6
H2 Dose#7
H2 Dose#8
H2 Dose#9
H2 Dose#10
H2 Dose#11
H2 Dose#12

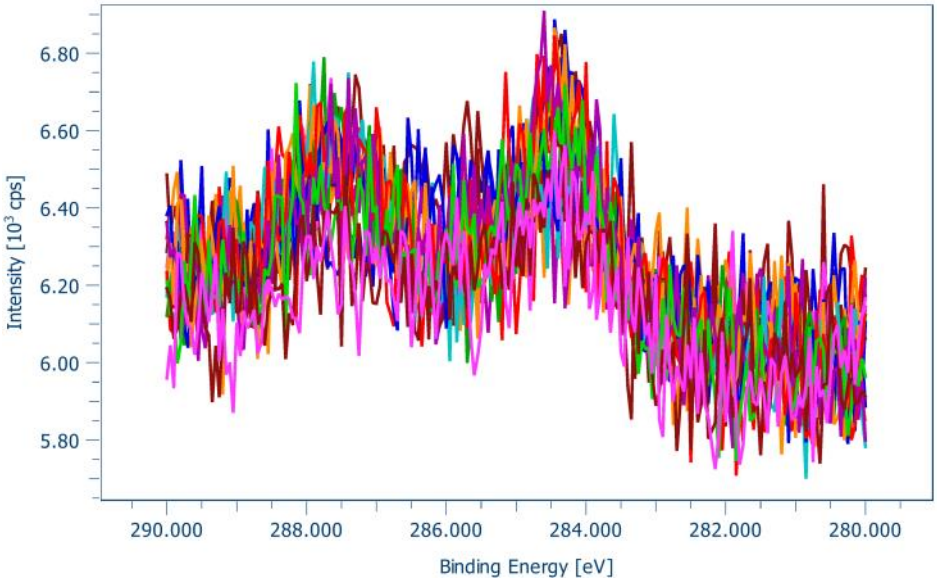
H2 Dose#13

~~H2 Dose#14~~ (not shown)









Connected new O2 tank
Pressure in first stage is 2700 psi at 4:00 PM
Turn up O3 at 4:10 PM
Ozone into HP cell 5:45 PM ~30mTorr

200208 - PdAg + 550 K O2 + H2 [3rd cycle]

Saturday, February 8, 2020 9:14 AM

Turn down O3 and off (9:15 AM)

Measure
XPS: Ag after 2nd reduction - 66 degrees
Pressure was ~7e-9

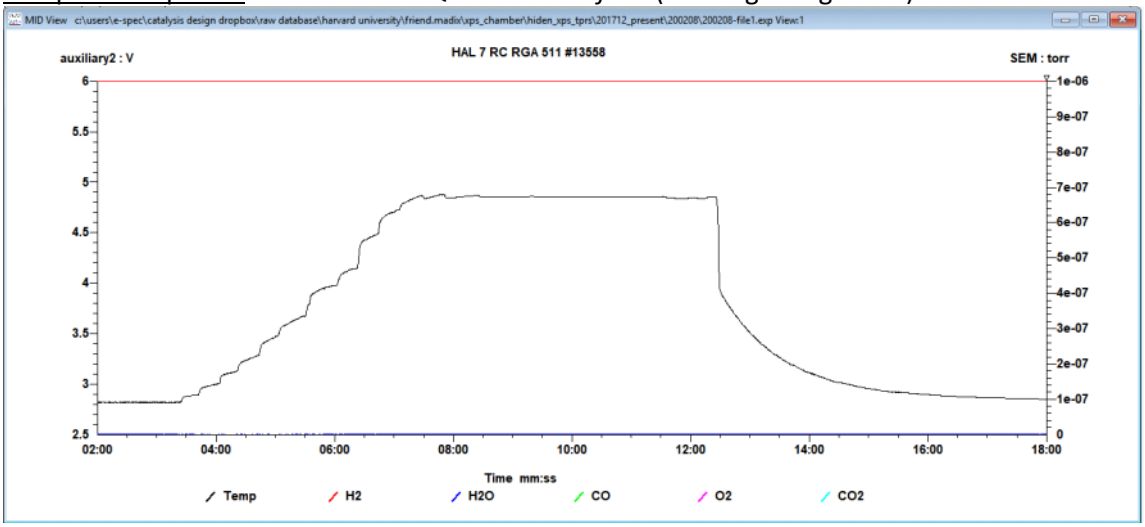
Need to leave 1 h 30 mins in UHV (at least till 10:45 AM) between turning down O3 and oxidizing

Connect cooling
10:30 AM

Turn up O2 at 11:00 AM

- **Oxidized** in 1.92(6) Torr O2 at 550 K for **5 min**
  - Valve cracked
  - O2 flowing from O3 conditions before
  - turbo 19 W
  - 1, 3, 5, 7, 10, 12, 15, 17, 20, 23, 24 W to ramp (in 20 sec intervals); let climb to 550 K at 24 W (started timer when it hit 550 K); 21.5 W laser power to stabilize at 550 K
  - maintained T within 550-555 K over duration of oxidation
  - Cooling in O2 till T = 305 K (~1 min) , then evacuating HP cell till p_baratron = 0.1 mTorr 11:31 AM

Temperature profile recorded with QMS: *200208-file1* (T at beginning 287 K)

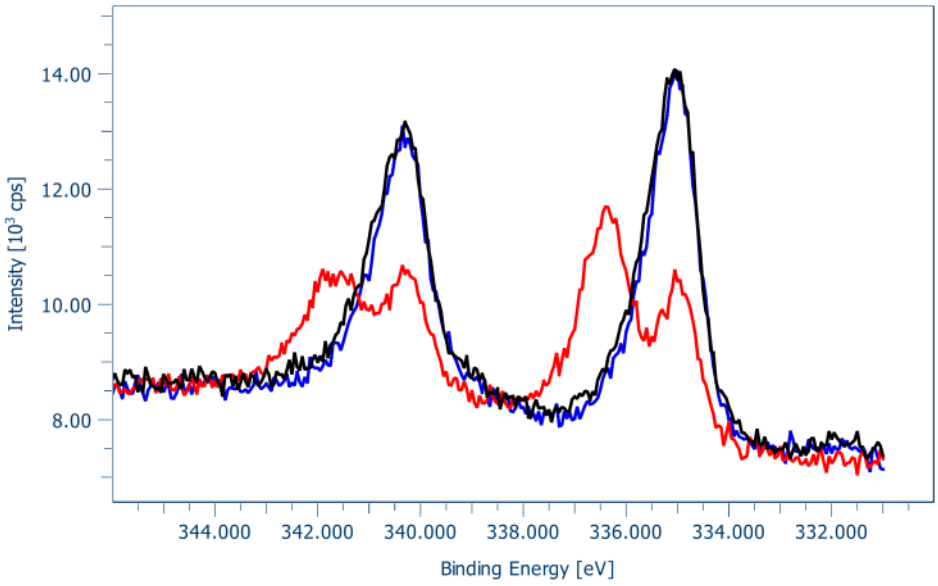


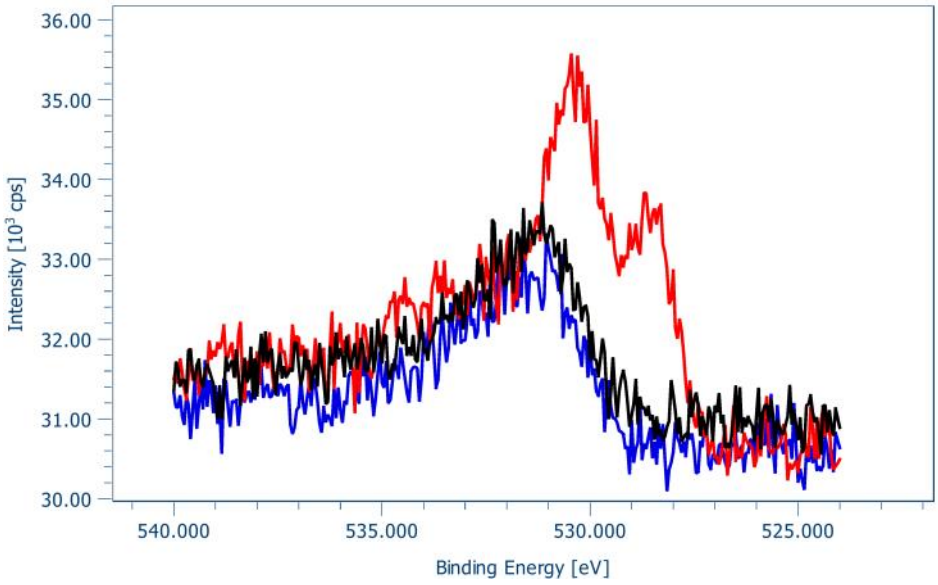
Switch back to air cooling for H2 dose; clear water from cryostat
Shut valve to HP cell at 15.0"
Turn on X-ray source at 15.0"

O2 pre-stage pressure was 2350 psi at 11:44 AM (lost 350 psi)
Closed O2 valves

Measure (11:39 AM)
XPS: after oxidation - 66 degrees

H2 Dose#14
Ag after 2nd reduction
After oxidation





SAME DOSES AS 1ST CYCLE

- **H2 Dose#1**: 1e-7 H2 for 1 min at 295 K (background, right leak valve)
  (5.2e-8 in prep; p_bkg = 7e-9 p_delta = 4.5e-8 ; had analysis ion gauge off)
  XPS: H2 Dose#1 - 66 degrees
  p_prep = 8.3e-9
  Done 12:05 PM

- **H2 Dose#2**: 1e-5 H2 for 1 min at 296 K (background, right leak valve)
  (5.0e-6 in prep; had analysis ion gauge off)
  XPS: H2 Dose#2 - 66 degrees
  p_prep = 1.0e-8
  Done 12:19 PM

- **H2 Dose#3**: 1e-4 H2 for 1 min at 296 K (background, right leak valve)
  (5.0e-5 in prep; had analysis ion gauge off)
  Wait 2.5 min to recover pressure
  XPS: H2 Dose#3 - 66 degrees
  p_prep = 2.1e-8
  Done 12:36 PM

- **H2 Dose#4**: 2e-4 H2 for 1 min (total: 1 min) at 297 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 2.5 min to recover pressure
  XPS: H2 Dose#4 - 66 degrees
  p_prep = 2.1e-8
  Done 12:54 PM

- **H2 Dose#5**: 2e-4 H2 for 1**.5** min (total: 2**.5** min) at 297 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 2.5 min to recover pressure
  Accidentally set timer wrong, did 1.5 min instead of 1 min. Balance out in next dose!
  XPS: H2 Dose#5 - 66 degrees
  p_prep = 2.3e-8
  Done 1:11 PM

- **H2 Dose#6**: 2e-4 H2 for 2**.5** min (total: 5 min) at 297 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#6 - 66 degrees
  p_prep = 2.1e-8
  Done 1:30 PM

- **H2 Dose#7**: 2e-4 H2 for 3 min (total: 8 min) at 297 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#7 - 66 degrees
  p_prep = 2.5e-8
  Done at 1:51 PM Turned off source 3 min later 1:54 PM
  Regime of autocatalytic reduction of PdO

- **H2 Dose#8**: 2e-4 H2 for 1 min (total: 9 min) at 297 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#8 - 66 degrees
  p_prep = 3.0e-8
  Done at 2:17 PM
  Regime of autocatalytic reduction of PdO

  Adjusted H2 leak valve, pressure now drops to ~2-3e-8 after 3 min wait after H2 dose and 1.0e-8 after scan

- **H2 Dose#9**: 2e-4 H2 for 1 min (total: 10 min) at 297 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#9 - 66 degrees

p_prep = 2.0e-8
Done at 2:35 PM
Regime of autocatalytic reduction of PdO

- **H2 Dose#10**: 2e-4 H2 for 5 min (total: 15 min) at 297 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#10 - 66 degrees
  p_prep = 1.9e-8
  Done at 3:01 PM

- **H2 Dose#11**: 2e-4 H2 for 5 min (total: 20 min) at 298 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#11 - 66 degrees
  p_prep = 2.1e-8
  Done at 3:24 PM

- **H2 Dose#12**: 2e-4 H2 for 10 min (total: 30 min) at 298 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#12 - 66 degrees
  p_prep = 2.2e-8
  Done at 3:55 PM

- **H2 Dose#13**: 2e-4 H2 for 10 min (total: 40 min) at 298 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#13 - 66 degrees
  p_prep = 2.5e-8
  Done at 4:23 PM
  Spectra overlapping with #12, but last dose for consistency

- **H2 Dose#14**: 2e-4 H2 for 10 min (total: 50 min) at 297 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#14 - 66 degrees
  p_prep =.e-8
  Done at PM
  Overlapping --> DONE!

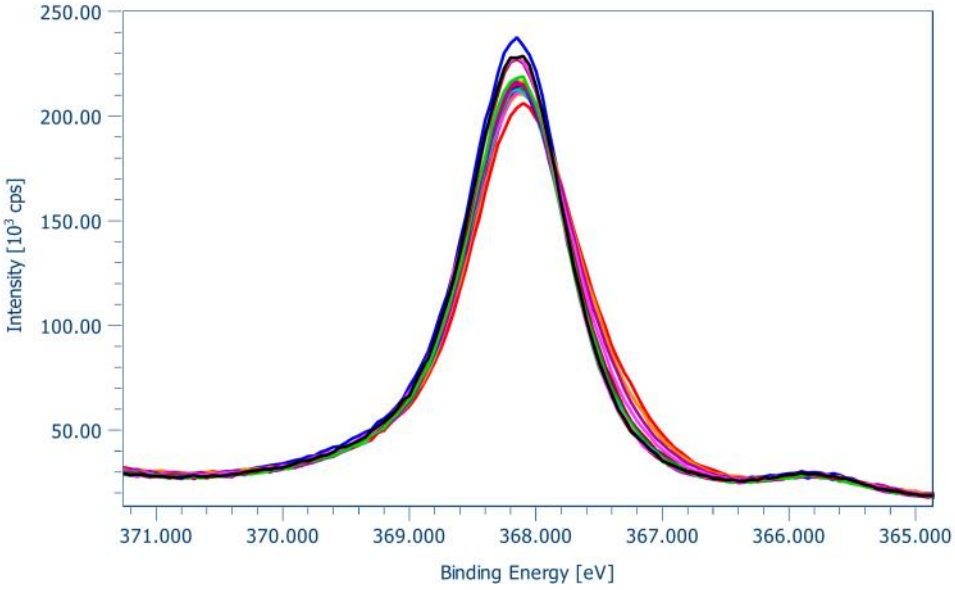
Don't forget VB spectrum!!

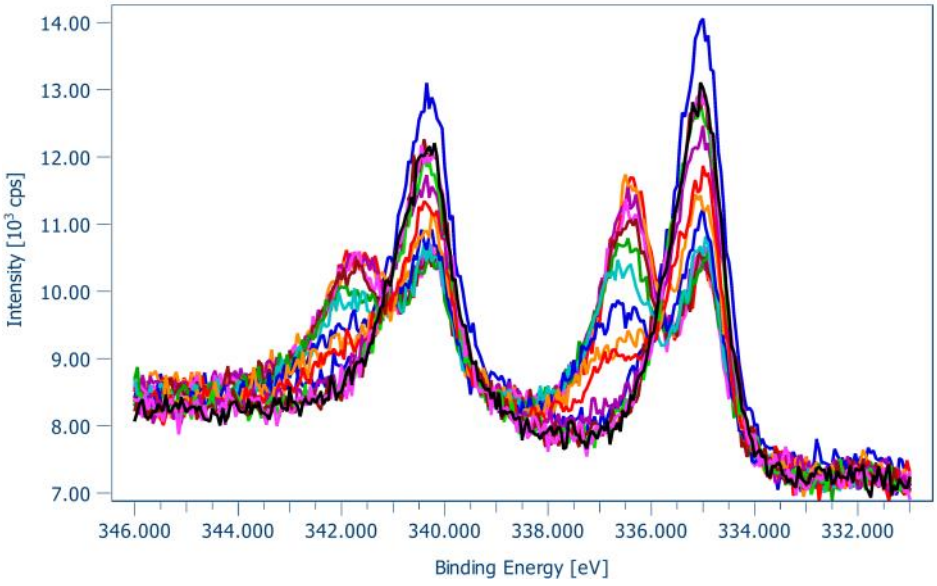
Background
0.01*5*60=3.0 L per pump-down
13*3 = 39 L per whole day

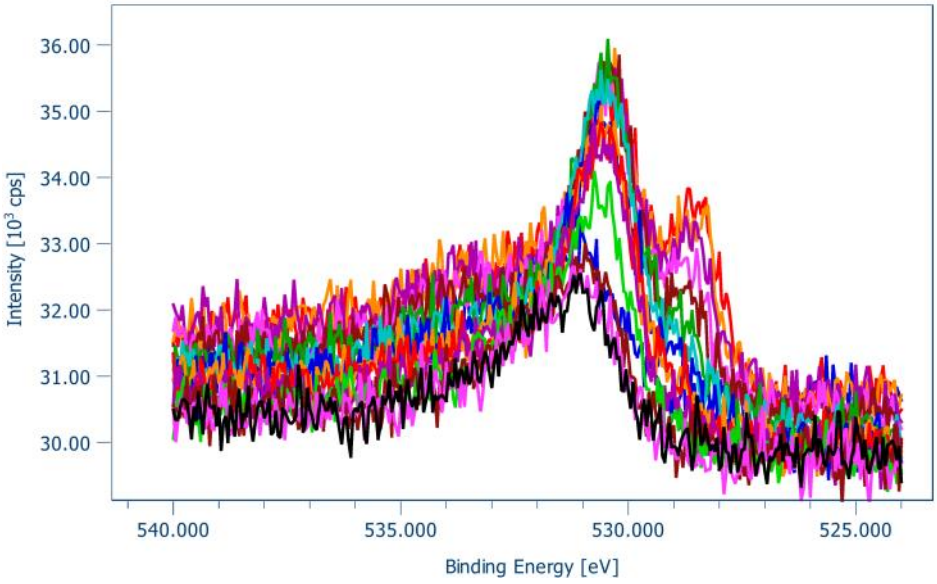
E-4 H2 Dose equivalent
100 L per second --> 39/100 = 0.39 sec for a E-4 Dose equates accumulated estimates background dose throughout the day in idle period

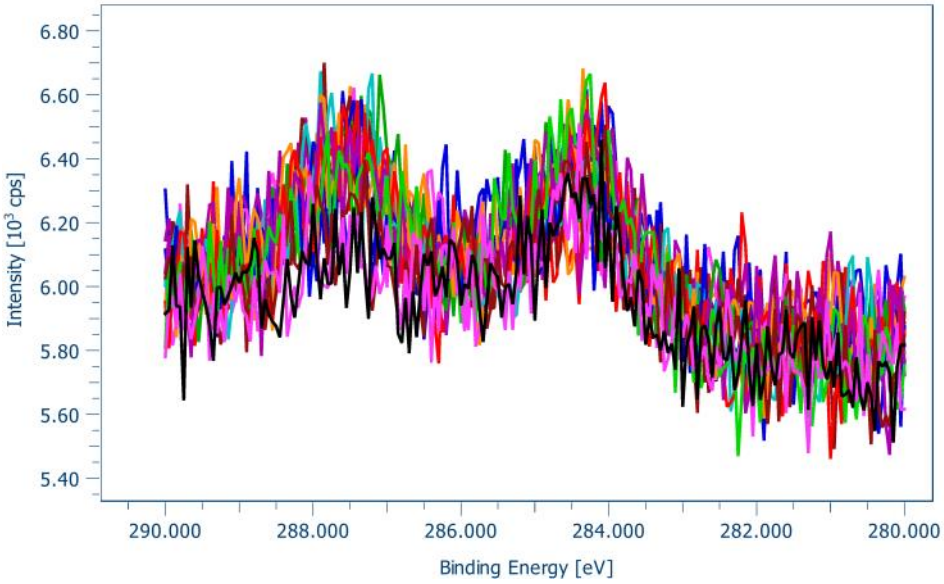
Legend:

H2 Dose#14 (day before)
After oxidation
H2 Dose#1
H2 Dose#2
H2 Dose#3
H2 Dose#4
H2 Dose#5
H2 Dose#6
H2 Dose#7
H2 Dose#8
H2 Dose#9
H2 Dose#10
H2 Dose#11
H2 Dose#12
H2 Dose#13
H2 Dose#14









Turned on O2 at 3:50 PM
Turn up O3 at 4:14 PM
Ozone into HP cell 5:18 PM ~30mTorr

200209 - PdAg + 550 K O2 + H2 [4th cycle]

Sunday, February 9, 2020 11:59 AM

p_prep = 3.6e-9 (background pressure after pumping down over night)

Turn down O3 and off (12:00 PM)
p_O2 = 2025 psi at 12 PM (lost ~375 psi since ~4 PM yday)

Measure
XPS: Ag after 3rd reduction - 66 degrees

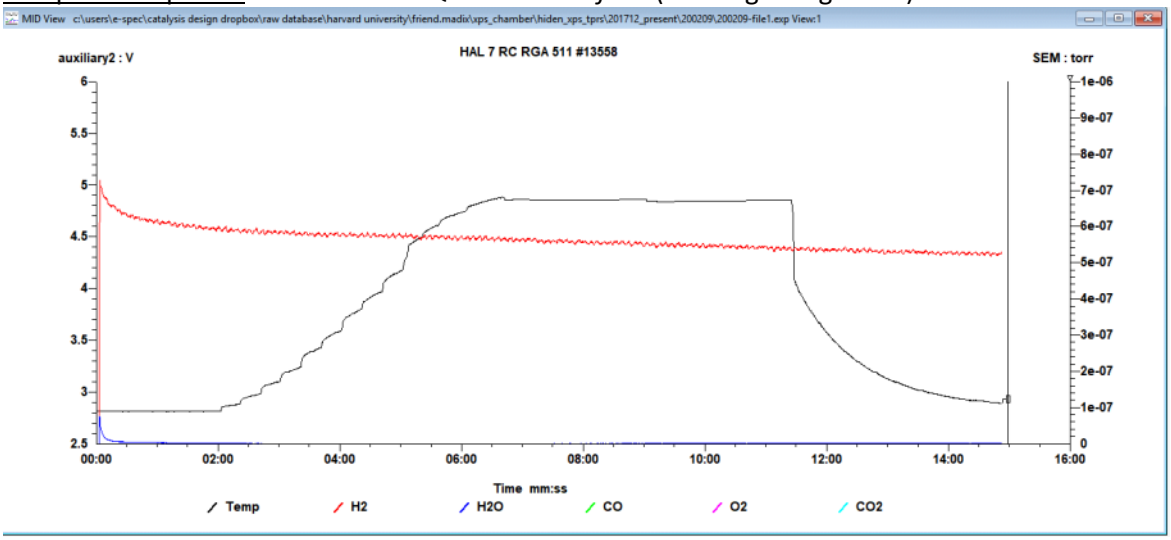
Need to leave 1 h 30 mins in UHV (at least till 1:30 PM) between turning down O3 and oxidizing

Connect cooling
2:02 PM

Turn up O2 at 2:30 PM

- **Oxidized** in 1.92(6) Torr O2 at 550 K for **5 min**
  - Valve cracked
  - O2 flowing from O3 conditions before
  - turbo 19 W
  - 1, 3, 5, 7, 10, 12, 15, 17, 20, 23, 24, 25 W to ramp (in 20 sec intervals); let climb to 550 K at 25 W (started timer when it hit 550 K); 21.5 W laser power to stabilize at 550 K
  - Cooling was much stronger this time
  - maintained T within 550-555 K over duration of oxidation
  - Cooling in O2 till T = 305 K (~1 min) , then evacuating HP cell till p_baratron = 0.1 mTorr 11:31 AM

Temperature profile recorded with QMS: *200209-file1* (T at beginning 285 K)



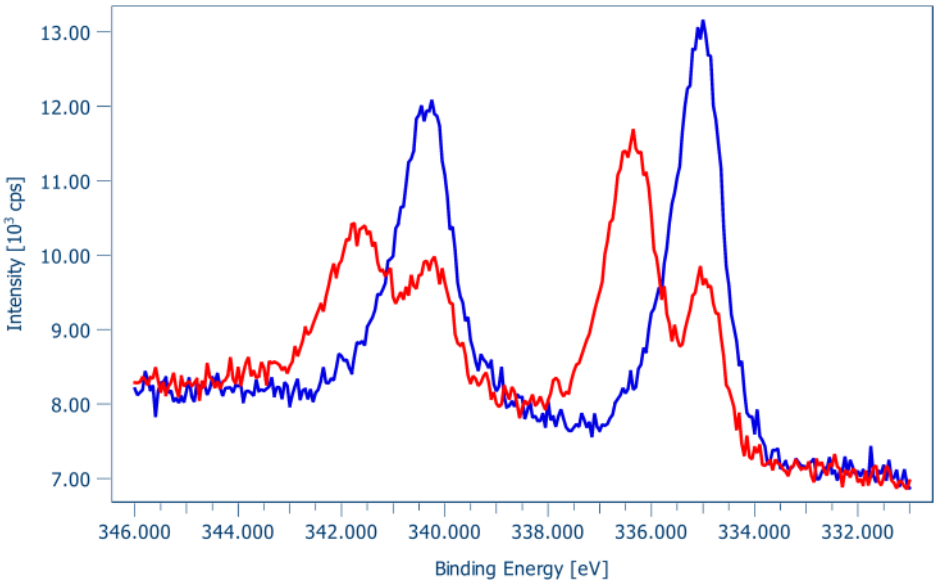
Switch back to air cooling for H2 dose; clear water from cryostat
Shut valve to HP cell at 15.0"
Turn on X-ray source at 15.0"

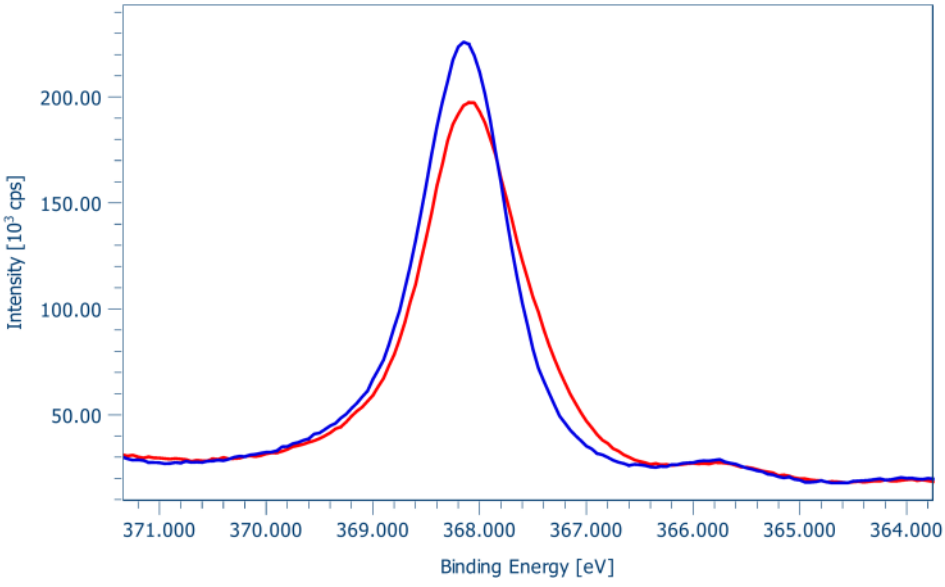
O2 pre-stage pressure was 1975 psi at 3:10 PM

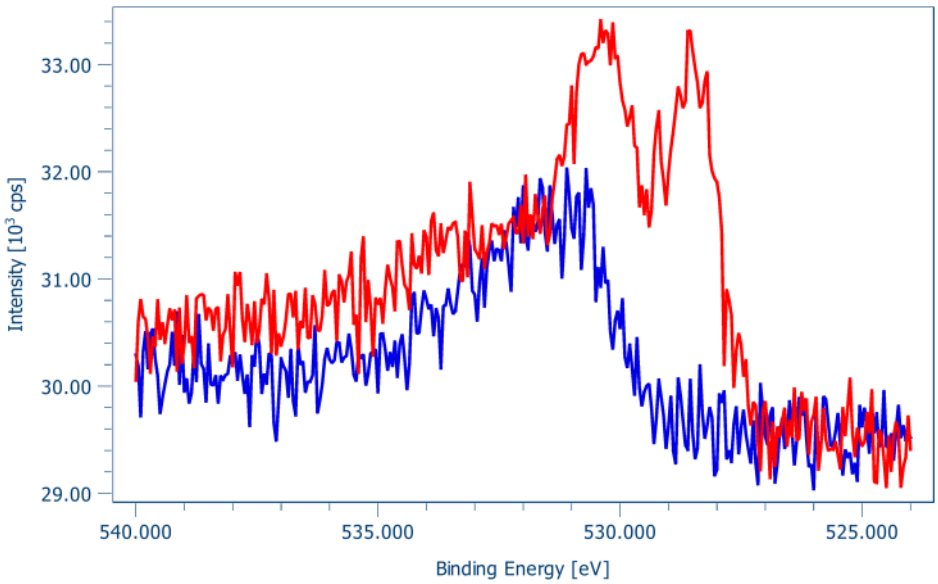
Closed O2 valves

Measure (3:09 PM)
XPS: after oxidation - 66 degrees

H2 Dose#14
Ag after 3rdreduction
After oxidation







SAME DOSES AS 1ST CYCLE

- **H2 Dose#1**: 1e-7 H2 for 1 min at 295 K (background, right leak valve)
  (4.9e-8 in prep; p_bkg = 3.6e-9 p_delta = 4.5e-8 ; had analysis ion gauge off)
  XPS: H2 Dose#1 - 66 degrees
  p_prep = 4.6e-9
  Done 3:33 PM

- **H2 Dose#2**: 1e-5 H2 for 1 min at 296 K (background, right leak valve)
  (5.0e-6 in prep; had analysis ion gauge off)
  XPS: H2 Dose#2 - 66 degrees
  p_prep = 7.5e-9
  Done 3:46 PM

- **H2 Dose#3**: 1e-4 H2 for 1 min at 296 K (background, right leak valve)
  (5.0e-5 in prep; had analysis ion gauge off)
  Wait 2.5 min to recover pressure
  XPS: H2 Dose#3 - 66 degrees
  p_prep = 2.0e-8
  Done 4:02 PM

- **H2 Dose#4**: 2e-4 H2 for 1 min (total: 1 min) at 297 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 2.5 min to recover pressure
  XPS: H2 Dose#4 - 66 degrees
  p_prep = 2.2e-8
  Done 4:19 PM

- **H2 Dose#5**: 2e-4 H2 for 1 min (total: 2 min) at 297 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 2.5 min to recover pressure
  XPS: H2 Dose#5 - 66 degrees
  p_prep = 2.0e-8
  Done 4:37 PM

- **H2 Dose#6**: 2e-4 H2 for 3 min (total: 5 min) at 297 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#6 - 66 degrees
    p_prep = 2.1e-8
    Done 4:58 PM

- **H2 Dose#7**: 2e-4 H2 for 3 min (total: 8 min) at 297 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#7 - 66 degrees
    p_prep = 2.2e-8
    Done at PM

- **H2 Dose#8**: 2e-4 H2 for 1 min (total: 9 min) at 297 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#8 - 66 degrees
    p_prep = 2.5e-8
    Done at 5:39 PM

- **H2 Dose#9**: 2e-4 H2 for 1 min (total: 10 min) at 297 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#9 - 66 degrees
    p_prep = 1.8e-8
    Done at 5:59 PM
    Regime of autocatalytic reduction of PdO?

- **H2 Dose#10**: 2e-4 H2 for 5 min (total: 15 min) at 297 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#10 - 66 degrees
    p_prep = 2.1e-8
    Done at 6:24 PM

  Probably start O2 flow again

- **H2 Dose#11**: 2e-4 H2 for 5 min (total: 20 min) at 298 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#11 - 66 degrees
    p_prep = 1.6e-8
    Done at 6:49 PM

- **H2 Dose#12**: 2e-4 H2 for 10 min (total: 30 min) at 298 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#12 - 66 degrees
    p_prep = 2.1e-8
    Done at 7:17 PM

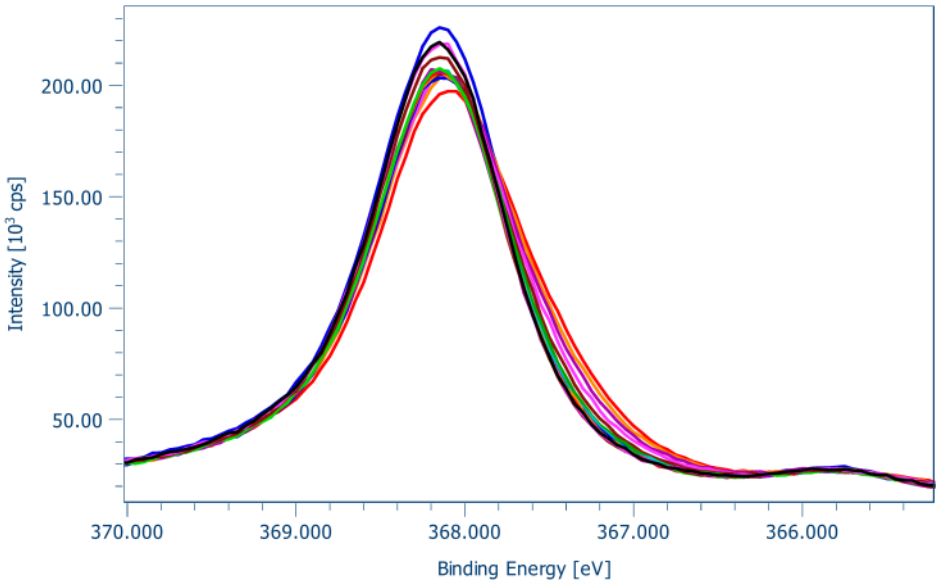
- **H2 Dose#13**: 2e-4 H2 for 10 min (total: 40 min) at 298 K (background, right leak valve)

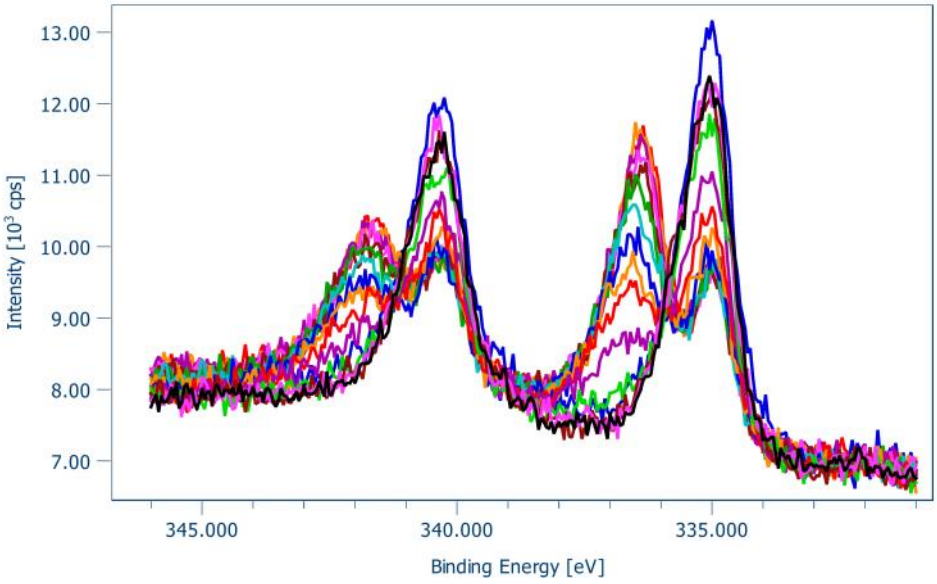
(1.0e-4 in prep; had analysis ion gauge off)
Wait 3 min to recover pressure
XPS: H2 Dose#13 - 66 degrees
p_prep = 2.2e-8
Done at 4:23 PM
Spectra overlapping with #12, but last dose for consistency

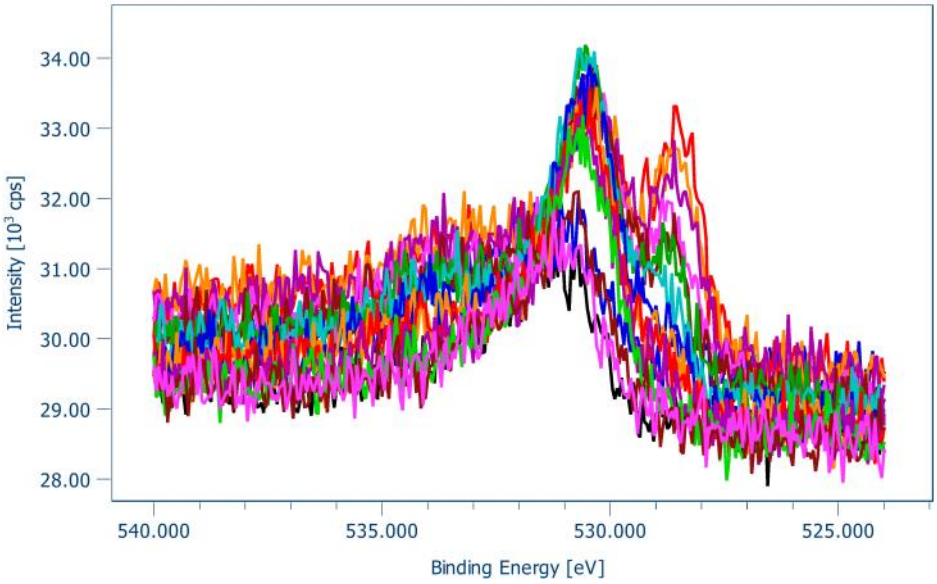
- **H2 Dose#14**: 2e-4 H2 for 10 min (<u>total: 50 min</u>) at 297 K (background, right leak valve)
(1.0e-4 in prep; had analysis ion gauge off)
Wait 3 min to recover pressure
XPS: H2 Dose#14 - 66 degrees
p_prep =2.0e-8
Done at PM
Overlapping --> DONE!

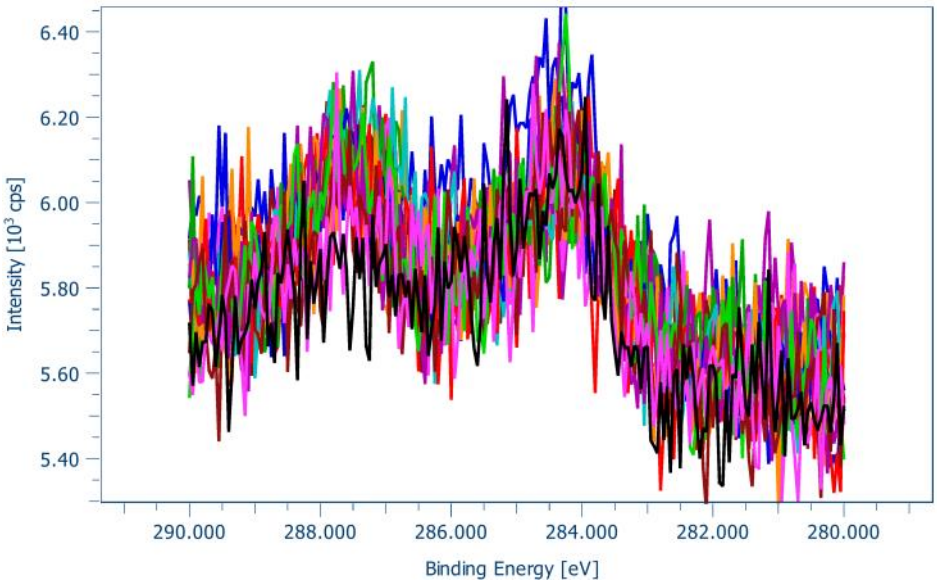
Don't forget VB spectrum!!

<u>Legend:</u>
~~**H2 Dose#14 (day before)**~~
Ag after 3rdreduction
After oxidation
H2 Dose#1
H2 Dose#2
H2 Dose#3
H2 Dose#4
H2 Dose#5
H2 Dose#6
H2 Dose#7
H2 Dose#8
H2 Dose#9
H2 Dose#10
H2 Dose#11
H2 Dose#12
H2 Dose#13
H2 Dose#14




13.00
12.00
11.00
10.00
9.00
8.00
7.00
Intensity [10³ cps]
345.000
340.000
335.000
Binding Energy [eV]


34.00
33.00
32.00
31.00
30.00
29.00
28.00
Intensity [10³ cps]
540.000
535.000
530.000
525.000
Binding Energy [eV]


6.40
6.20
6.00
5.80
5.60
5.40
Intensity [10³ cps]
290.000
288.000
286.000
284.000
282.000
280.000
Binding Energy [eV]

Turned on O2 at 5:50 PM
Turn up O3 at 6:25 PM
Ozone into HP cell 8:25 PM ~30mTorr

200210 - PdAg + 550 K O2 + H2 [5th cycle]

Monday, February 10, 2020 9:19 AM

p_prep = 2.4e-9 (background pressure after pumping down over night)

Turn down O3 and off (9:20 AM)
p_O2 = 1750 psi at 9:20 AM (lost ~225 psi since ~9 PM yday)

Measure
XPS: Ag after 4th reduction - 66 degrees

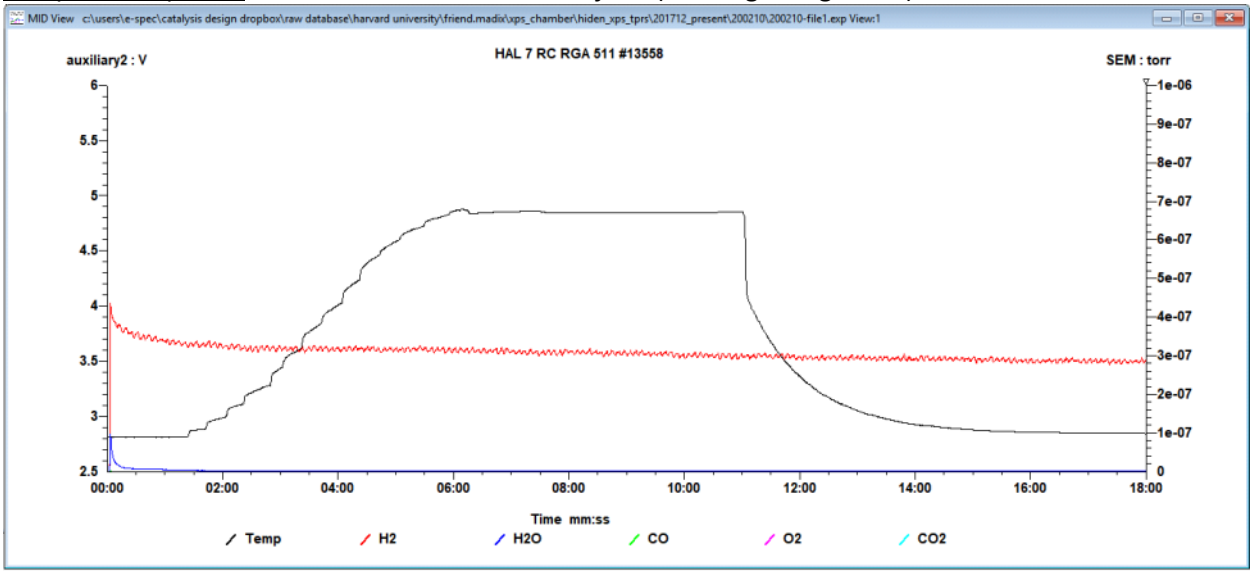
Need to leave 1 h 30 mins in UHV (at least till 10:50 AM) between turning down O3 and oxidizing

Connect cooling
10:30 AM

Turn up O2 at 11:24 AM

- **Oxidized** in 1.93(1) Torr O2 at 550 K for **5 min**
  - Valve cracked
  - O2 flowing from O3 conditions before
  - turbo 19 W
  - 1, 3, 5, 7, 10, 12, 15, 17, 20, 23, 24, 25 W to ramp (in 20 sec intervals); let climb to 550 K at 25 W (started timer when it hit 550 K); 23.5 W laser power to stabilize at 550 K
  - maintained T within 550-555 K over duration of oxidation
  - Cooling in O2 till T = 305 K (~1 min) , then evacuating HP cell till p_baratron = 0.1 mTorr 11:50 AM

Temperature profile recorded with QMS: *200210-file1* (T at beginning 286 K)



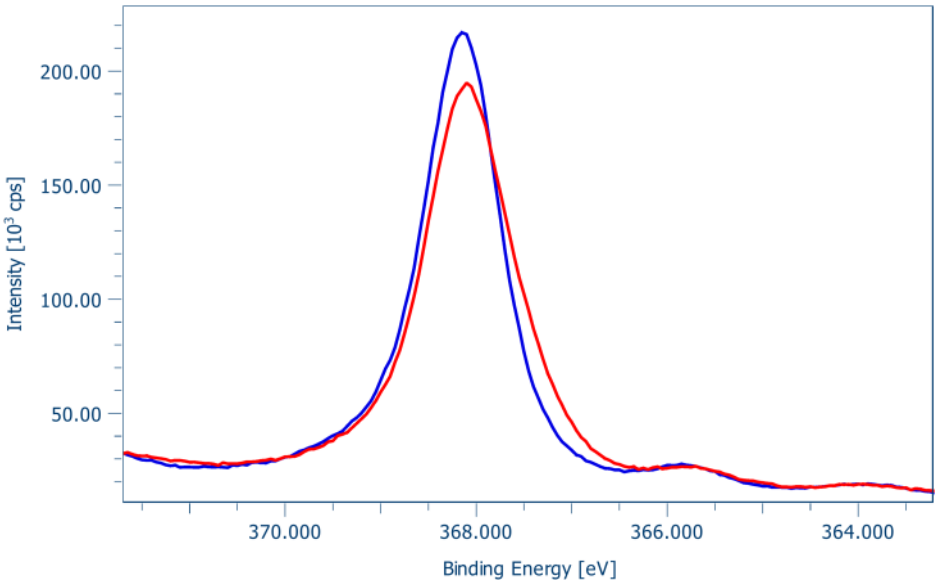
Switch back to air cooling for H2 dose; clear water from cryostat
Shut valve to HP cell at 15.0"
Turn on X-ray source at 15.0"

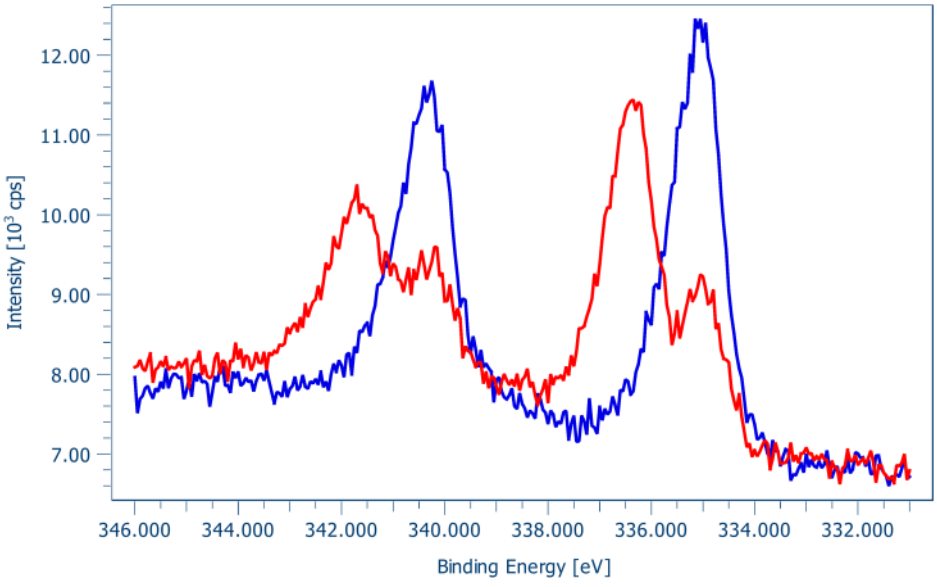
O2 pre-stage pressure was 1700 psi at 12:04 PM

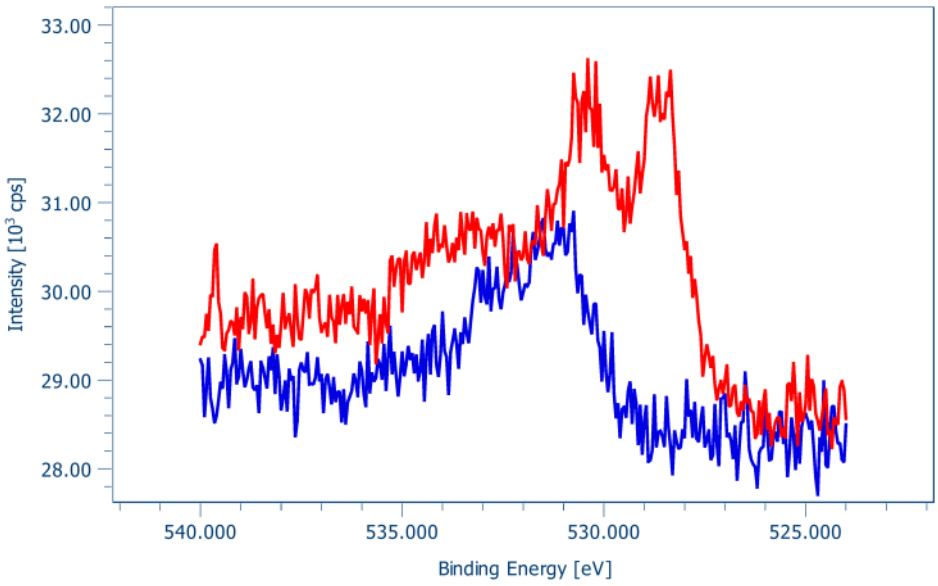
Closed O2 valves

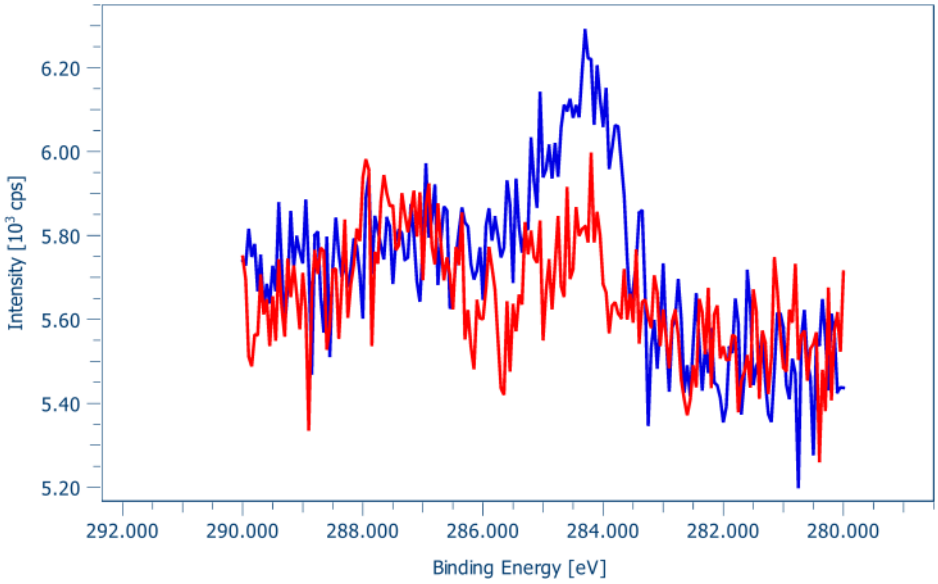
Measure (12:01 PM)
XPS: after oxidation - 66 degrees

H2 Dose#14
Ag after 2nd reduction
After oxidation









SAME DOSES AS 1ST CYCLE

- **H2 Dose#1**: 1e-7 H2 for 1 min at 295 K (background, right leak valve)
  (4.8e-8 in prep; p_bkg = 2.7e-9 p_delta = 4.5e-8 ; had analysis ion gauge off)
  XPS: H2 Dose#1 - 66 degrees
  p_prep = 3.8e-9
  Done 12:23 PM

- **H2 Dose#2**: 1e-5 H2 for 1 min at 296 K (background, right leak valve)
  (5.0e-6 in prep; had analysis ion gauge off)
  XPS: H2 Dose#2 - 66 degrees
  p_prep = 6.4e-9
  Done 12:38 PM

- **H2 Dose#3**: 1e-4 H2 for 1 min at 296 K (background, right leak valve)
  (5.0e-5 in prep; had analysis ion gauge off)
  Wait 2.5 min to recover pressure

XPS: H2 Dose#3 - 66 degrees
p_prep = 1.9e-8
Done 12:54 PM

- **H2 Dose#4**: 2e-4 H2 for 1 min (total: 1 min) at 297 K (background, right leak valve)
(1.0e-4 in prep; had analysis ion gauge off)
Wait 2.5 min to recover pressure
XPS: H2 Dose#4 - 66 degrees
p_prep = 1.9e-8
Done 1:11 PM

- **H2 Dose#5**: 2e-4 H2 for 1 min (total: 2 min) at 297 K (background, right leak valve)
(1.0e-4 in prep; had analysis ion gauge off)
Wait 2.5 min to recover pressure
XPS: H2 Dose#5 - 66 degrees
p_prep = 2.1e-8
Done 1:28 PM

- **H2 Dose#6**: 2e-4 H2 for 3 min (total: 5 min) at 297 K (background, right leak valve)
(1.0e-4 in prep; had analysis ion gauge off)
Wait 3 min to recover pressure
XPS: H2 Dose#6 - 66 degrees
p_prep = 2.2e-8
Done 1:47 PM

- **H2 Dose#7**: 2e-4 H2 for 3 min (total: 8 min) at 297 K (background, right leak valve)
(1.0e-4 in prep; had analysis ion gauge off)
Wait 3 min to recover pressure
XPS: H2 Dose#7 - 66 degrees
p_prep = 1.8e-8
Done at 2:06 PM

- **H2 Dose#8**: 2e-4 H2 for 1 min (total: 9 min) at 297 K (background, right leak valve)
(1.0e-4 in prep; had analysis ion gauge off)
Wait 3 min to recover pressure
XPS: H2 Dose#8 - 66 degrees
p_prep = 2.0e-8
Done at 2:23 PM

- **H2 Dose#9**: 2e-4 H2 for 1 min (total: 10 min) at 297 K (background, right leak valve)
(1.0e-4 in prep; had analysis ion gauge off)
Wait 3 min to recover pressure
XPS: H2 Dose#9 - 66 degrees
p_prep = 1.9e-8
Done at 2:39 PM
~Regime of autocatalytic reduction of PdO

- **H2 Dose#10**: 2e-4 H2 for 5 min (total: 15 min) at 297 K (background, right leak valve)
(1.0e-4 in prep; had analysis ion gauge off)
Wait 3 min to recover pressure
XPS: H2 Dose#10 - 66 degrees
p_prep =1.9 e-8
Done at 3:01 PM

~Regime of autocatalytic reduction of PdO

- **H2 Dose#11**: 2e-4 H2 for 5 min (<u>total: 20 min</u>) at 297 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#11 - 66 degrees
  p_prep = 2.1e-8
  Done at 3:23 PM

- **H2 Dose#12**: 2e-4 H2 for 10 min (<u>total: 30 min</u>) at 297 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#12 - 66 degrees
  p_prep = 2.1e-8
  Done at 3:51 PM

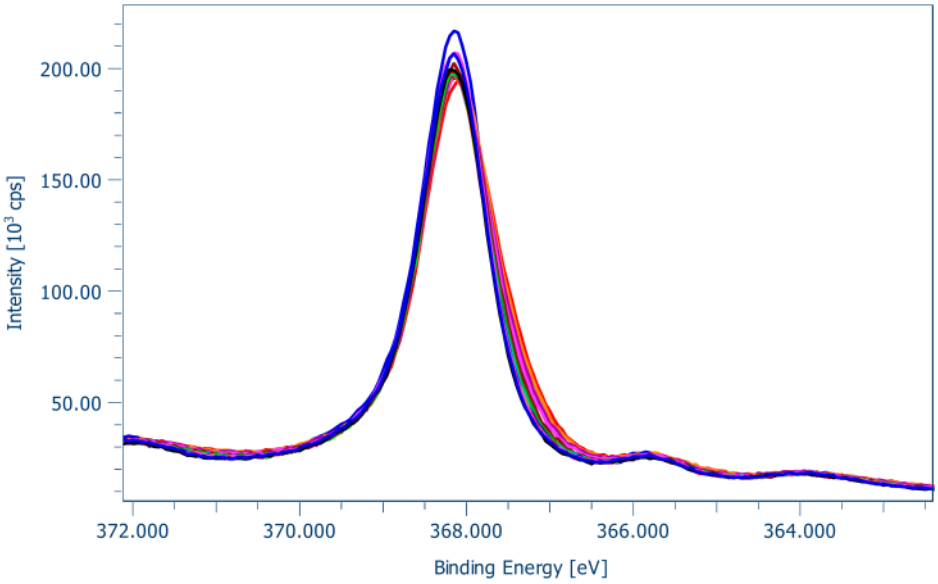
- **H2 Dose#13**: 2e-4 H2 for 10 min (<u>total: 40 min</u>) at 297 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#13 - 66 degrees
  p_prep = 2.2e-8
  Done at 4:19 PM

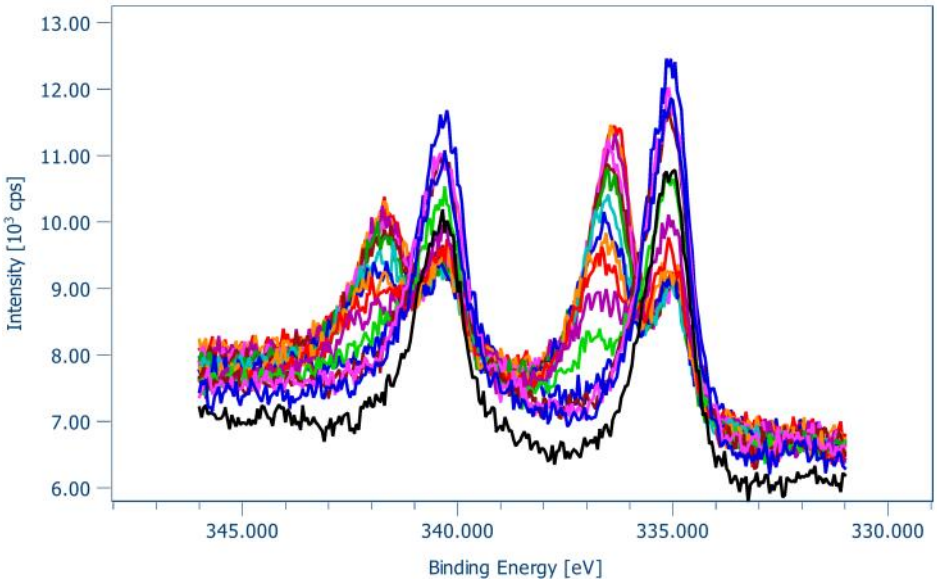
- **H2 Dose#14**: 2e-4 H2 for 10 min (<u>total: 50 min</u>) at 297 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#14 - 66 degrees
  p_prep =1.7e-8
  Done at 4:51 PM
  Background lower for some reason (what happened??), do one more dose and measure to check if spectra are overlapping
  Don't forget VB spectrum!!

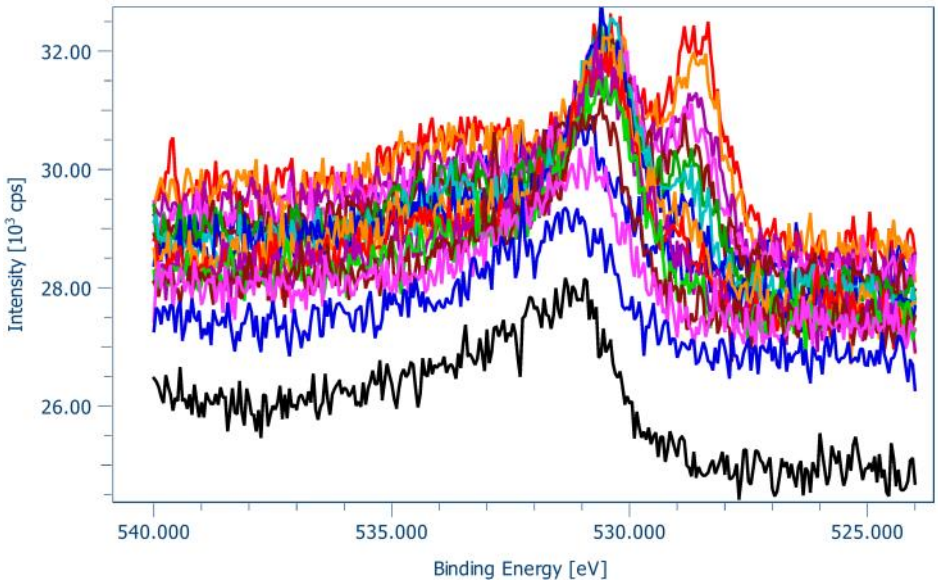
- **H2 Dose#15**: 2e-4 H2 for 10 min (<u>total: 60 min</u>) at 297 K (background, right leak valve)
  (1.0e-4 in prep; had analysis ion gauge off)
  Wait 3 min to recover pressure
  XPS: H2 Dose#15 - 66 degrees
  p_prep = 2.3e-8
  Done at PM
  Basically overlapping spectra compared to H2 Dose#13 but still small baseline offset (more visible at higher BE e.g. for O1s)
  Something with H2 Dose#14 wasn't right and the baseline was offset. Should probably use Dose#15 and replace with #14 (total dosing time of 50 min)
  Don't forget VB spectrum!!

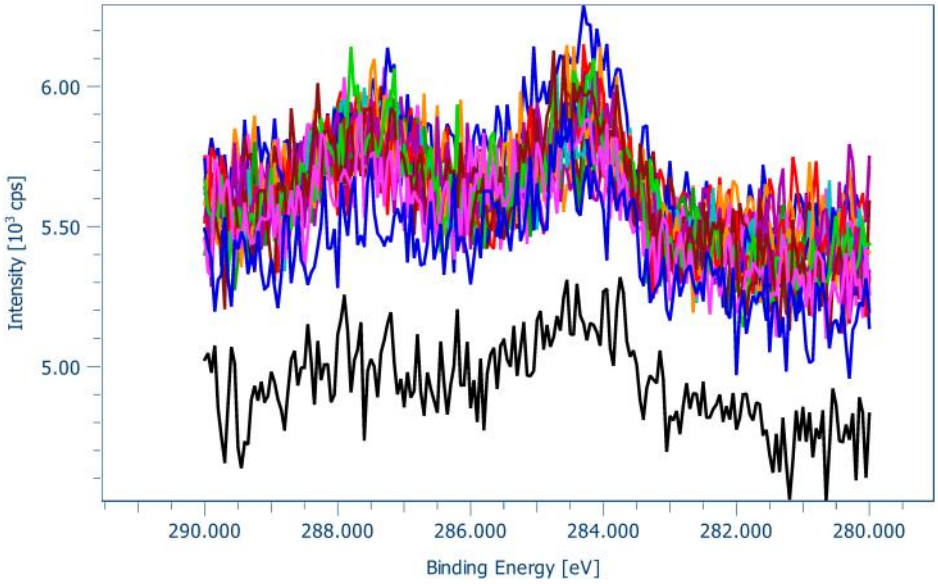
<u>Legend:</u>
~~**H2 Dose#14 (day before)**~~
Ag after 4th reduction
After oxidation
H2 Dose#1
H2 Dose#2
H2 Dose#3

H2 Dose#4
H2 Dose#5
H2 Dose#6
H2 Dose#7
H2 Dose#8
H2 Dose#9
H2 Dose#10
H2 Dose#11
H2 Dose#12
H2 Dose#13
H2 Dose#14
H2 Dose#15









Turned on O2 at 4:22 PM
Turn up O3 at 4:55 PM
Ozone into HP cell 6:15 PM ~30mTorr

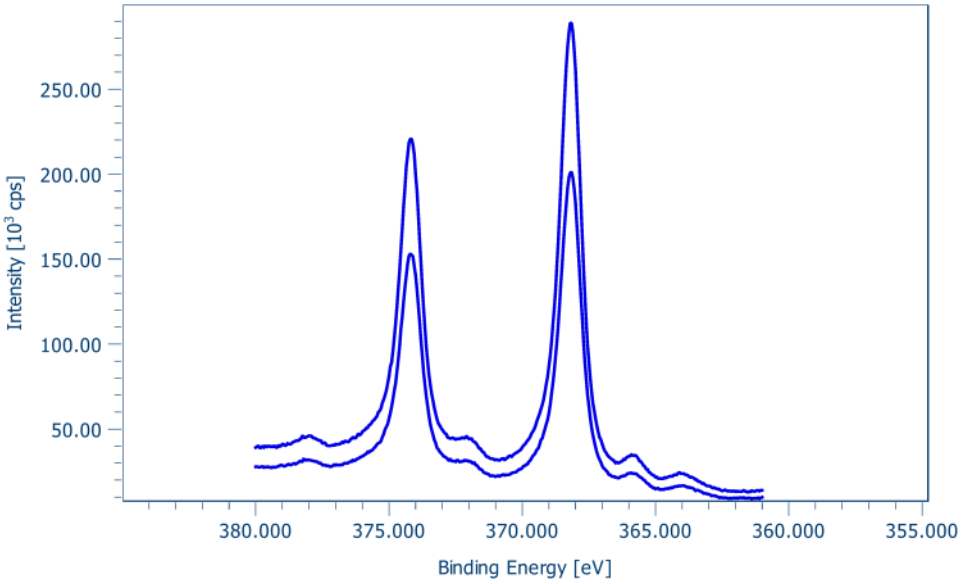
200218 - Ag + 550 K O2 [control]

Tuesday, February 18, 2020 9:51 AM

Turn down O3 and off (9:30 AM)
p_O2 = 1400 psi at 10:00 AM

Measure
XPS: Ag after sputter - 66 degrees

Detector must have suffered some loss over the past experiments :/



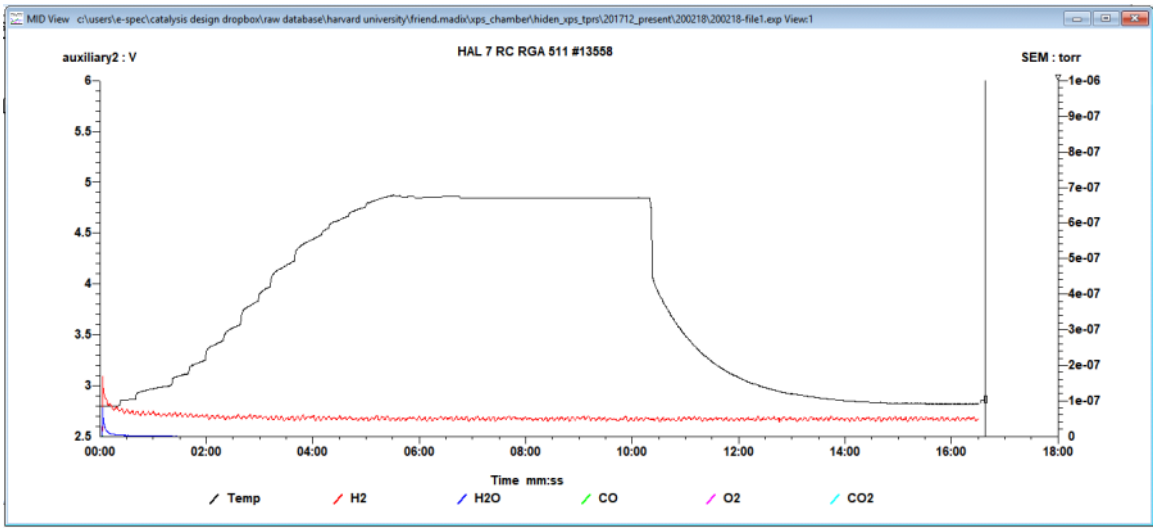
Need to leave 1 h 30 mins in UHV (at least till 11:00 AM) between turning down O3 and oxidizing

Connect cooling
11:10 AM

Turn up O2 at 11:20 AM

- **Oxidized** in 1.93(0) Torr O2 at 550 K for **5 min**
  - Valve cracked
  - O2 flowing from O3 conditions before
  - turbo 19 W
  - 1, 3, 5, 7, 10, 12, 15, 17, 20, 23, 24, 25, 26,27, 28 W to ramp (in 20 sec intervals); let climb to 550 K at 28 W (started timer when it hit 550 K); 27 W laser power to stabilize at 550 K
  - Cooling pretty strong this time, therefore higher laser power
  - maintained T within 550-555 K over duration of oxidation
  - Cooling in O2 till T = 305 K (~1 min) , then evacuating HP cell till p_baratron = 0.1 mTorr 11: AM

Temperature profile recorded with QMS: *200218-file1* (T at beginning 284 K)

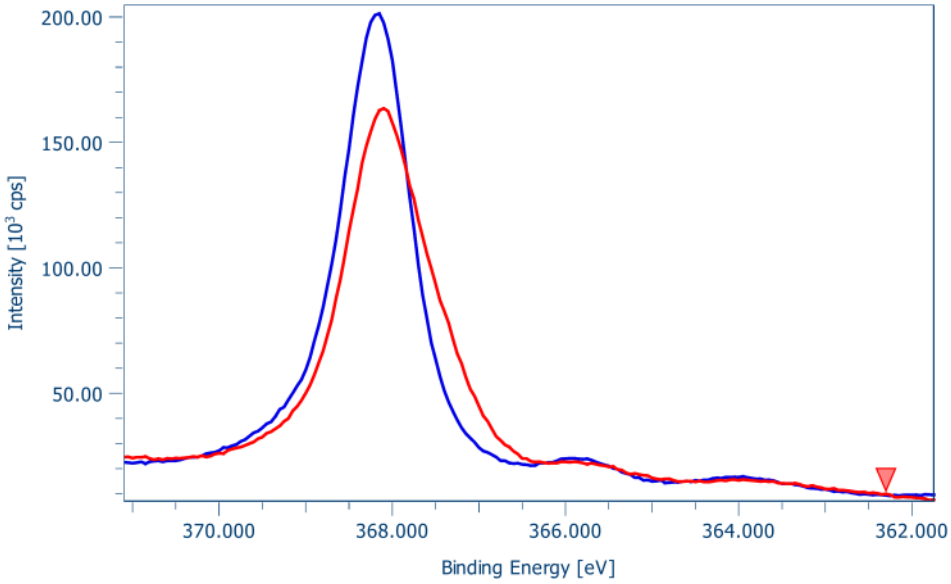


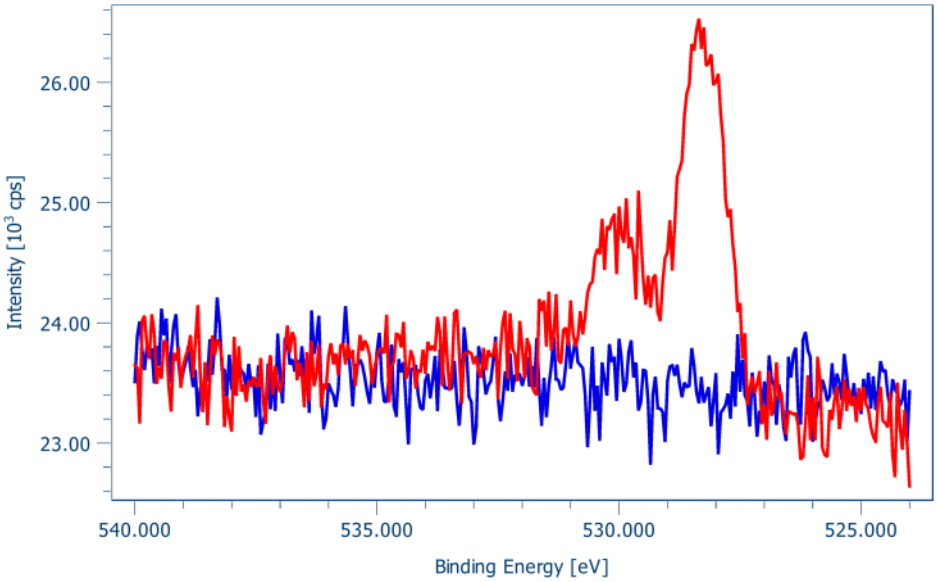
Switch back to air cooling for H2 dose; clear water from cryostat
Shut valve to HP cell at 15.0"
Turn on X-ray source at 15.0"

O2 pre-stage pressure was 1350 psi at 12:00 PM
Closed O2 valves

Measure (12:00 PM)
XPS: after oxidation - 66 degrees

Ag after sputter
After oxidation





SAME DOSES AS 1ST CYCLE but don't dose H2

- **H2 Dose#1**: 1 min at 295 K
  (had analysis ion gauge off)
  XPS: H2 Dose#1 - 66 degrees
  p_prep = 1.6e-9
  Done 12:22 PM

- **H2 Dose#2**: 1 min at 296 K
  (had analysis ion gauge off)
  XPS: H2 Dose#2 - 66 degrees
  p_prep = 1.5e-9
  Done 12:34 PM

- **H2 Dose#3**: 1 min at 296 K
  (had analysis ion gauge off)
  XPS: H2 Dose#3 - 66 degrees
  p_prep = 1.5e-9
  Done 12:45 PM

- **H2 Dose#4**: 1 min (total: 1 min) at 297 K
  (had analysis ion gauge off)
  XPS: H2 Dose#4 - 66 degrees
  p_prep = 1.5e-9
  Done 12:57 PM

- **H2 Dose#5**: 1 min (total: 2 min) at 297 K
  (had analysis ion gauge off)
  XPS: H2 Dose#5 - 66 degrees
  p_prep = 1.5e-9
  Done 1:08 PM

- **H2 Dose#6**: 3 min (total: 5 min) at 297 K
  (had analysis ion gauge off)

XPS: H2 Dose#6 - 66 degrees
p_prep = 1.4e-9
Done 1:25 PM

- **H2 Dose#7**: 3 min (total: 8 min) at 297 K
(had analysis ion gauge off)
XPS: H2 Dose#7 - 66 degrees
p_prep = 1.3e-9
Done at 1:40 PM

- **H2 Dose#8**: 1 min (total: 9 min) at 297 K
(had analysis ion gauge off)
XPS: H2 Dose#8 - 66 degrees
p_prep = 1.4e-9
Done at 1:53 PM

- **H2 Dose#9**: 1 min (total: 10 min) at 297 K
(had analysis ion gauge off)
XPS: H2 Dose#9 - 66 degrees
p_prep = 1.3e-9
Done at 2:06 PM

- **H2 Dose#10**: 5 min (total: 15 min) at 297 K
(had analysis ion gauge off)
XPS: H2 Dose#10 - 66 degrees
p_prep =1.3e-9
Done at 2:22 PM

- **H2 Dose#11**: 5 min (total: 20 min) at 297 K
(had analysis ion gauge off)
XPS: H2 Dose#11 - 66 degrees
p_prep = 1.3e-9
Done at 2:40 PM

- **H2 Dose#12**: 10 min (total: 30 min) at 297 K
(had analysis ion gauge off)
XPS: H2 Dose#12 - 66 degrees
p_prep = 1.3e-9
Done at 3:05 PM

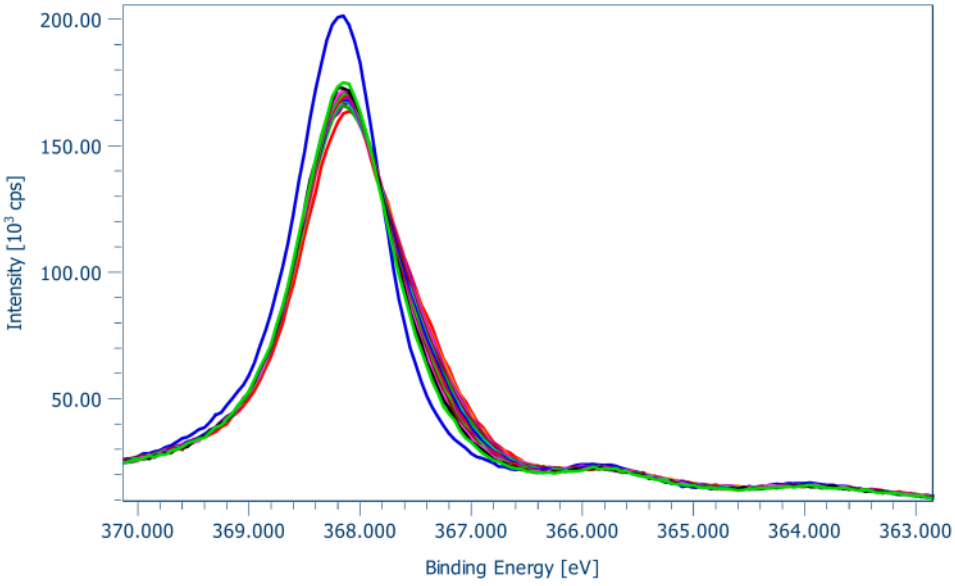
- **H2 Dose#13**: 10 min (total: 40 min) at 297 K
(had analysis ion gauge off)
XPS: H2 Dose#13 - 66 degrees
p_prep = 1.3e-9
Done at 3:29 PM

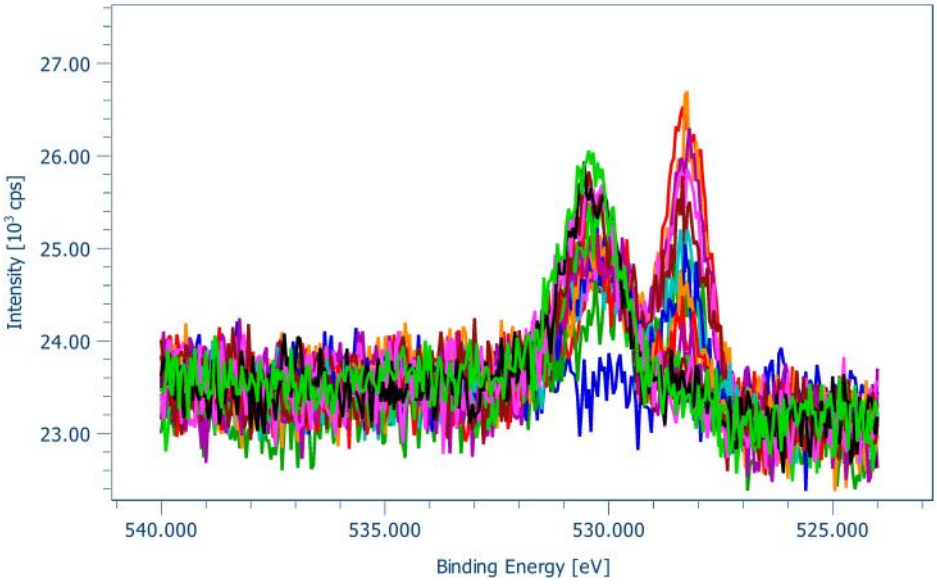
- **H2 Dose#14**: 10 min (total: 50 min) at 297 K
(had analysis ion gauge off)
XPS: H2 Dose#14 - 66 degrees
p_prep = 1.3e-9
Done at 3:55 PM

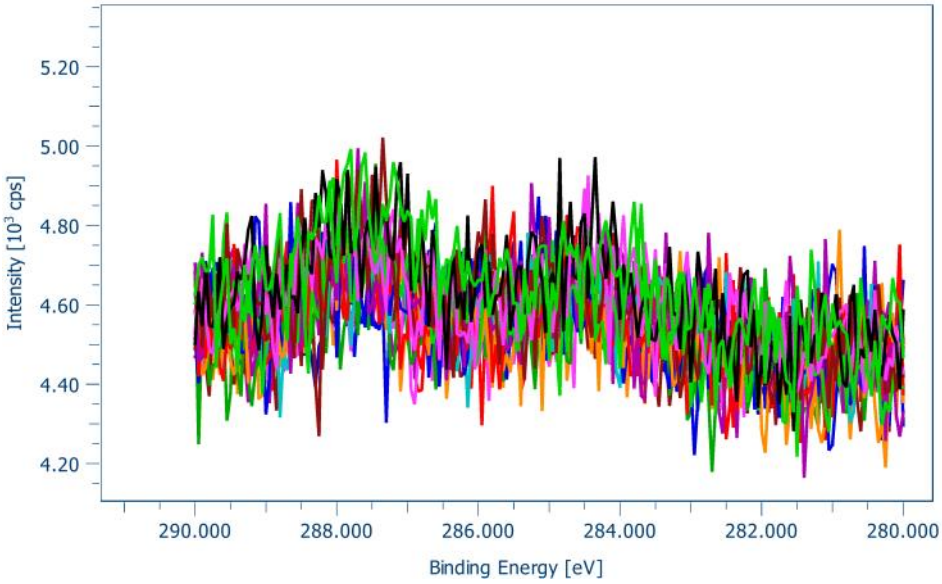
- **H2 Dose#15**: 10 min (total: 50 min) at 297 K

(had analysis ion gauge off)
XPS: H2 Dose#15 - 66 degrees
p_prep = 1.3e-9
Done at 4:22 PM

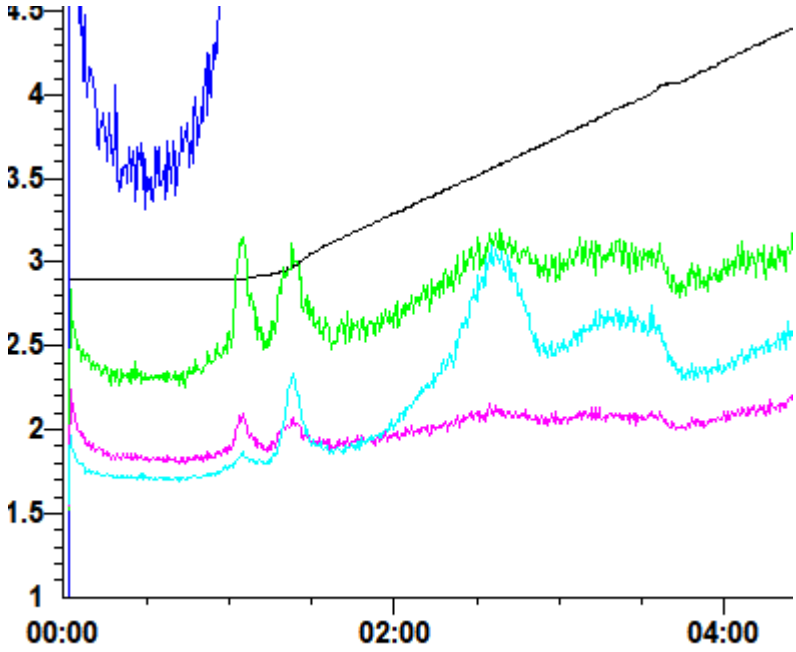
Legend:
Ag after sputter
After oxidation
H2 Dose#1
H2 Dose#2
H2 Dose#3
H2 Dose#4
H2 Dose#5
H2 Dose#6
H2 Dose#7
H2 Dose#8
H2 Dose#9
H2 Dose#10
H2 Dose#11
H2 Dose#12
H2 Dose#13
H2 Dose#14
H2 Dose#15







- **TPD**: 200218-file2
  Some CO2 around 400 K --> from Ag carbonate
  Rest is contribution from Cu / sample holder it seems at > 600 K


4.5
4
3.5
3
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2
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