Optimizing the Electrical Stability of Platinum Films Deposited in Lesker-Sputter

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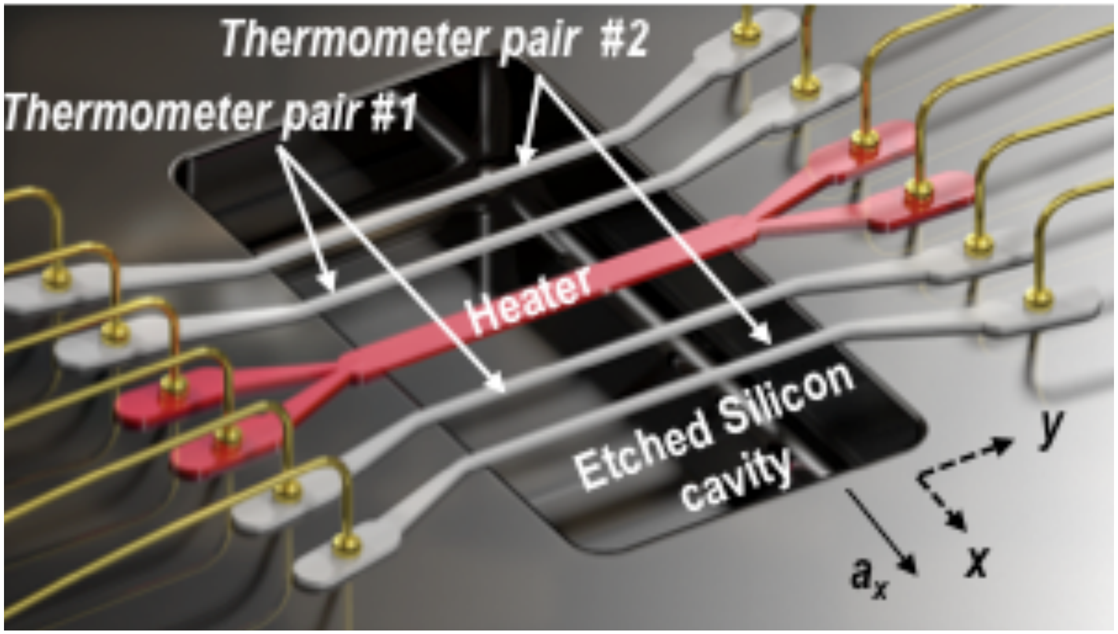
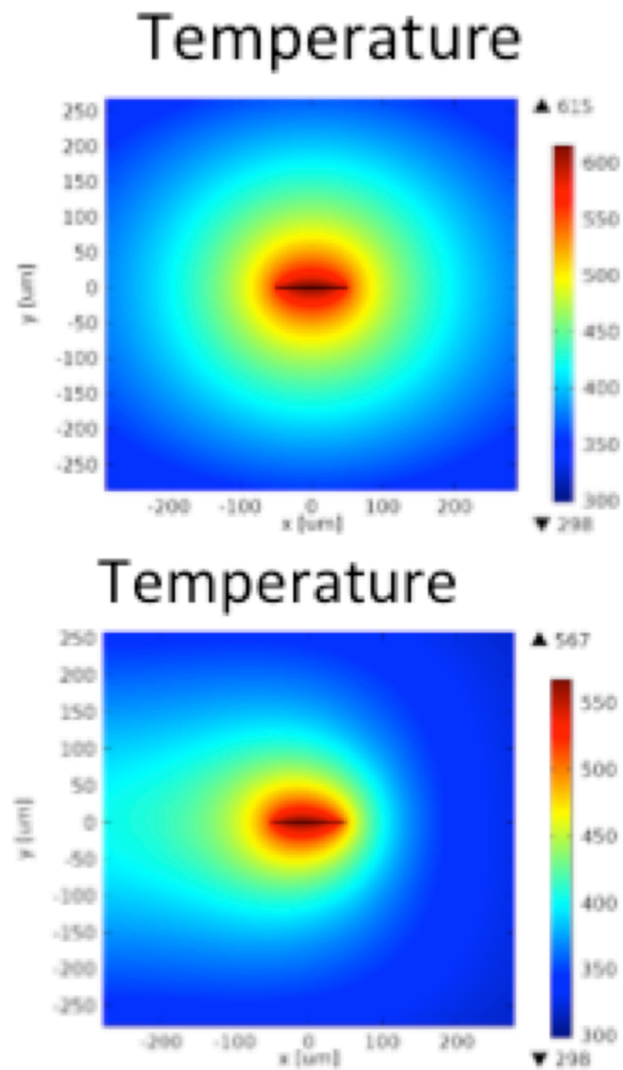
Motivation: Thermal Accelerometer





- Central heater creates hot air "bubble"
- Acceleration causes bubble to move i.e. asymmetry in temperature distribution
- Measurable ΔT between thermistors at equal distance from heater
- No movable parts
- Logarithmic sensitivity scaling

Thermal Accelerometer Requirements

- Must be able to sense very small (<0.001 °C) temperature differences
- $\mathrm{TCR} = \dfrac{1}{\rho}\dfrac{\mathrm{d}\rho}{\mathrm{d}T}$ [ppm/°C] Resistance Stability $= \dfrac{\Delta\rho}{\rho}$ [ppm]
- Figure of Merit: Resistance Stability / TCR [°C]
- Mattheissen's Rule: $\rho = \rho_l(T) + \rho_d$
- Simplified Figure of Merit: $\Delta\rho$ $[\mu\Omega\cdot\mathrm{cm}]$ (assume $\dfrac{\mathrm{d}\rho_l}{\mathrm{dT}} = \mathrm{const}$)

Metrics for this project

Metric	Phase 1	Phase 2	Phase 3
ALD Resistance and TCR Stability	0.1% over 1 month, 100 cycles -40 to +80	50ppm over 1 month, 1K cycles -40 to +80	5ppm over 1 month, 10K cycles -40 to +80
Bias Stability	1000 µg @1S	100 µg @1S	30 µg @1S
Bias Stability	1000 µg @1000S	100 µg @1000S	10 µg @1000S
Scale Factor Repeatability	100 ppm	10 ppm	1 ppm
Bandwidth	100 Hz	1kHz	5kHz
Resolution (@1S)	1000 µg	100µg	10µg

Why Sputtering?

Pt Bulk Resistivity: 10.4 uOhm*cm

Resistivity from Standard Recipe Deposition:

ALD	e-beam Evaporation	Sputtering
14 uOhm*cm	17 uOhm*cm	20 uOhm*cm

Tunable Process Parameters:

ALD	e-beam Evaporation	Sputtering
Temperature [C]	Beam Current [mA]	Power [W]
Exposure Mode?	Sweep Speed?	Pressure [mTorr]
		Temperature [C]
		Substrate Bias [V]
		DC vs. RF Power

Only in Lesker Sputter!

Experimental Approach – DOE

Number	DC/RF	Power	Pressure	Temperature	Substrate Bias
1	DC	-	-	-	-
2	DC	-	-	+	+
3	DC	-	+	+	+
4	DC	+	-	+	-
5	DC	+	+	-	-
6	DC	+	+	-	+

Number	DC/RF	Power	Pressure	Temperature	Substrate Bias
7	RF	-	-	-	-
8	RF	-	+	-	+
9	RF	-	+	+	-
10	RF	+	-	-	+
11	RF	+	-	+	+
12	RF	+	+	+	-

Low / high values:

- Power
  Low: 70W DC, 40W RF
  High: 4x Low
- Pressure
  Low: 2 mT
  High: 30 mT
- Temperature
  Low: no heating
  High: 270 C
- Substrate Bias
  Low: 0 W
  High: 40 W (~125 V)

1) For each parameter set, do test deposition to determine growth rate
2) Using that rate, aim for 30 nm thickness on oxidized Si wafer

Experimental Results

Number	DC/RF	Power	Pressure	Temperature	Substrate Bias	ρ [μΩ*cm]	Δρ [μΩ*cm] (RMS)
1	DC	-	-	-	-	19.6	2.3E-04
2	DC	-	-	+	+	13.8	6.3E-04
3	DC	-	+	+	+	14.5	6.8E-04
4	DC	+	-	+	-	15.6	1.5E-04
5	DC	+	+	-	-	94.1	2.9E-04
6	DC	+	+	-	+	41.6	1.7E-03

Number	DC/RF	Power	Pressure	Temperature	Substrate Bias	ρ [μΩ*cm]	Δρ [μΩ*cm] (RMS)
7	RF	-	-	-	-	15.3	4.7E-04
8	RF	-	+	-	+	/	/
9	RF	-	+	+	-	13.1	5.7E-04
10	RF	+	-	-	+	15.9	7.6E-04
11	RF	+	-	+	+	14.7	5.3E-04
12	RF	+	+	+	-	12.1	4.2E-04

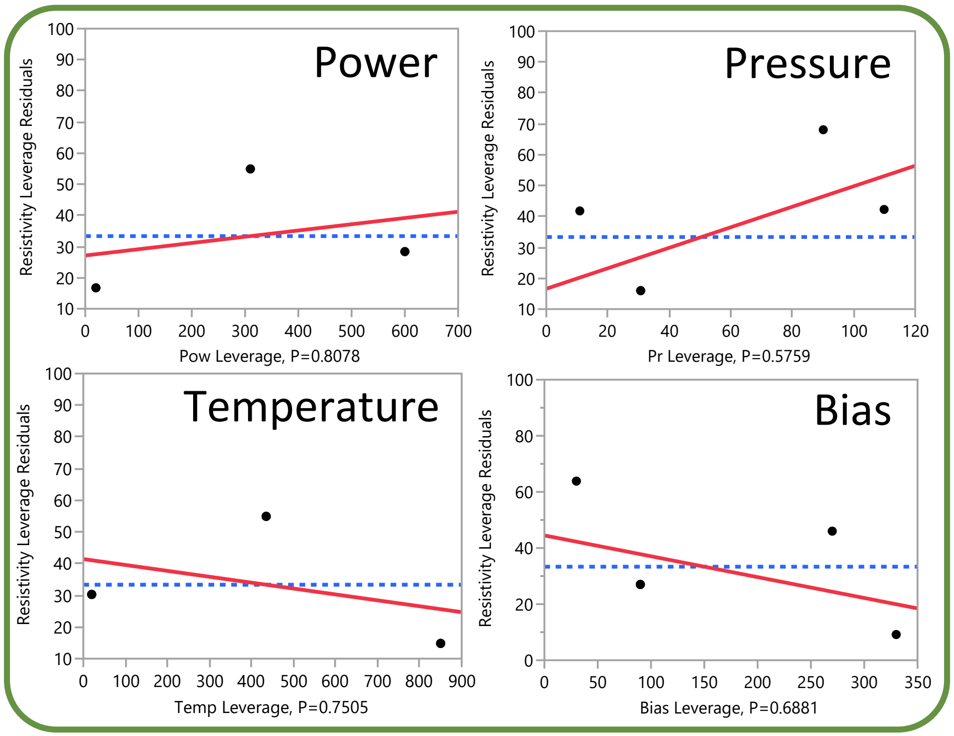
Bulk Platinum (Literature Value):	10.4 μΩ*cm	
Best ALD Film:	14.1 μΩ*cm	2.0E-04 μΩ*cm RMS
Evaporated Film (intlvac_evap, 100 nm):	17.1 μΩ*cm	

Experimental Results – Statistical Analysis

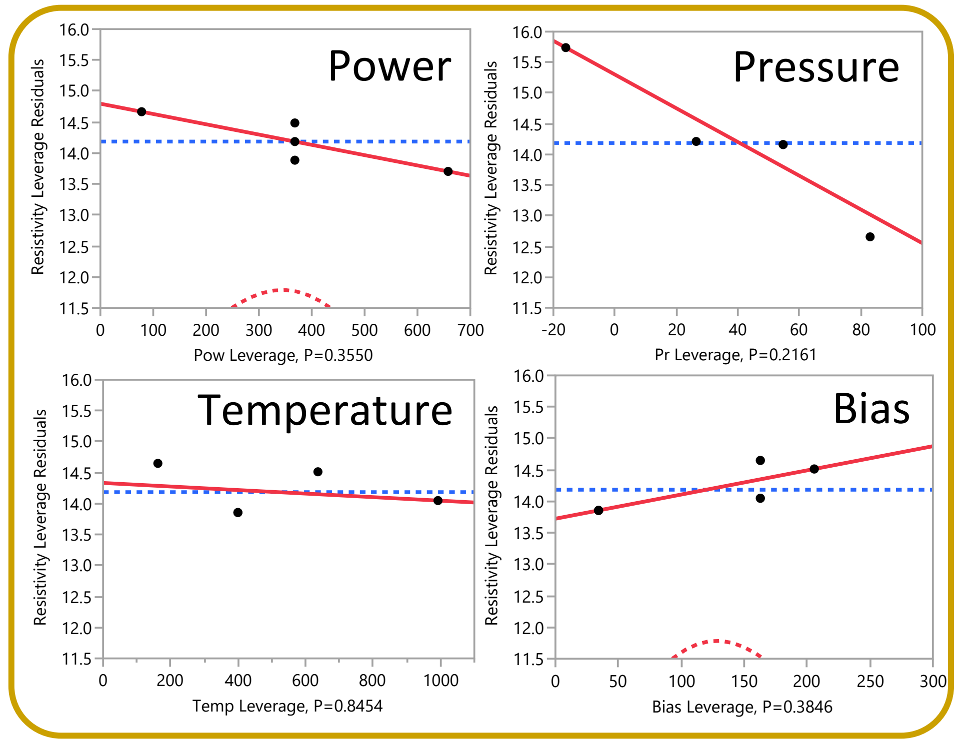
Source	LogWorth
Temp	0.800
PT	0.649
Pr	0.642
Pow	0.584
Bias	0.250

- Temperature most important parameter for resistivity, then power type – higher temperature and RF are better
- DC: lower power, lower pressure, higher bias better
- RF: higher power, higher pressure, lower bias better

DC

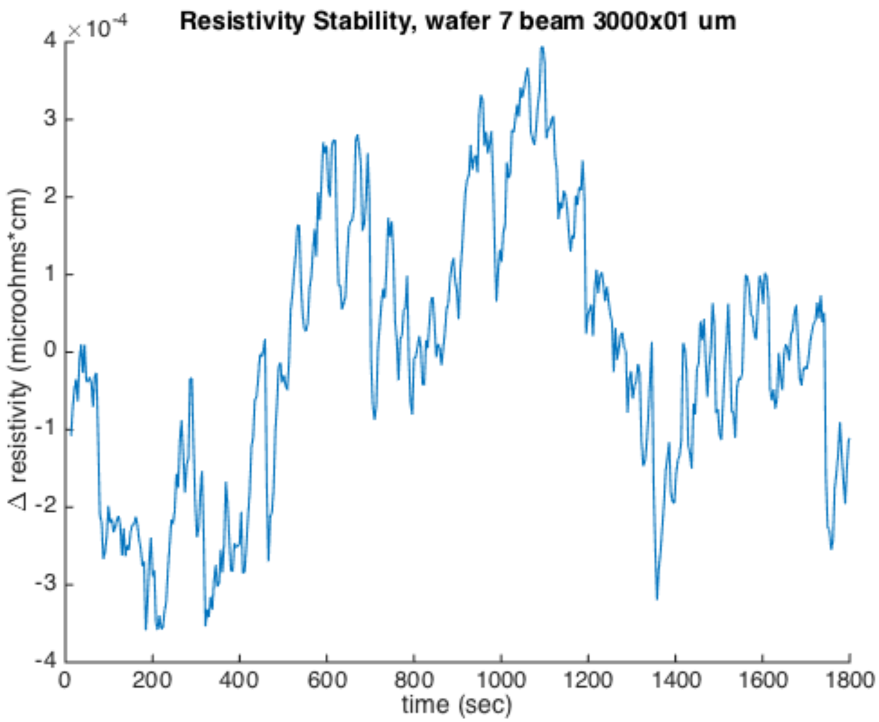


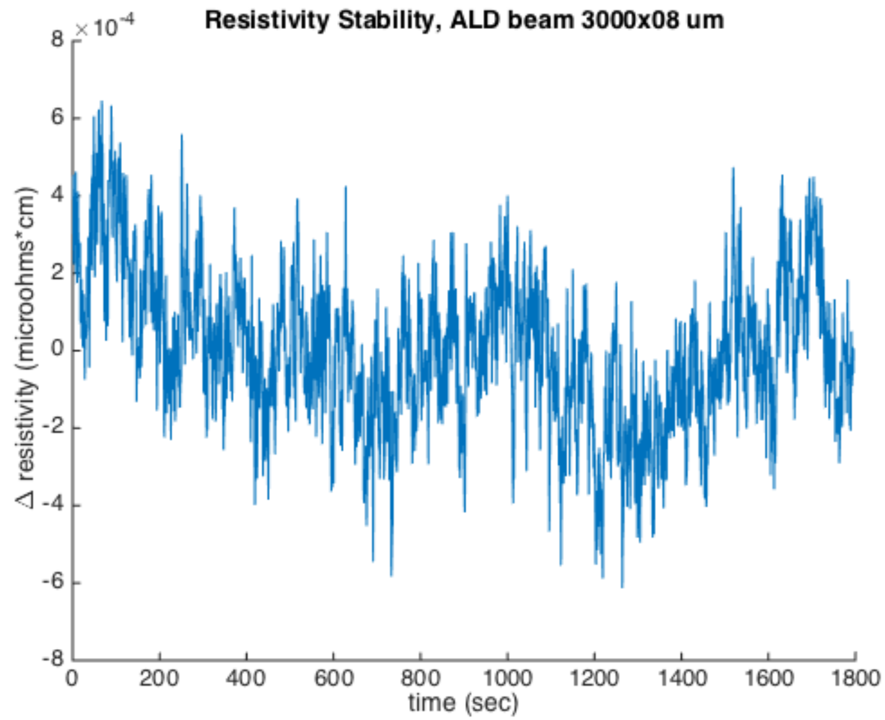
RF



Experimental Results – Stability Plots

- Lithographically patterned Pt beams, 0.5-10 um x 1000-8080 um
- 4-wire probed
- Resistance measurement ~ 1x per second for 30 min





Future Work

Electrical Testing

- Longer term resistivity stability measurements, with aggressive burn-in phase
- Analyze impact of thermistor dimensions
- Full TCR characterization

Morphology Characterization

- Redeposition of select recipes on (111) Si to enable XRD crystallinity analysis
- AFM characterization of film roughness

Further Recipe Development

- Iterate around best parameter set – try different temperatures and biases
- Test best condition on sapphire wafer – 0.6% lattice mismatch with (111) Pt

Process Integration

- Can substitute deposition method in Thermal Accelerometer Fabrication Process, but need to test adhesion – ALD probably better

Part 2: Lessons Learned

Process and Characterization Debugging

Lesker Sputter

Recipe Writing

- Existing Recipes can do Deposition, Heating and Biasing, but not at the same time, and they usually fail at plasma striking
- Manually activating heater or bias while running regular deposition recipe NOT advised – will likely trigger interlocks and cause trouble if aborted
- Developed our own "E241 Ultimate Master Recipe" that can do everything

Substrate Heating

- Max. 800 C, but even at 600 C awful base pressure (9E-8 -> 3E-5 Torr) resulting in oxidized film: 15% O in XPS after etching
- At 400 C, no oxidation but will form PtSi on Si substrate
- PID gains are way off

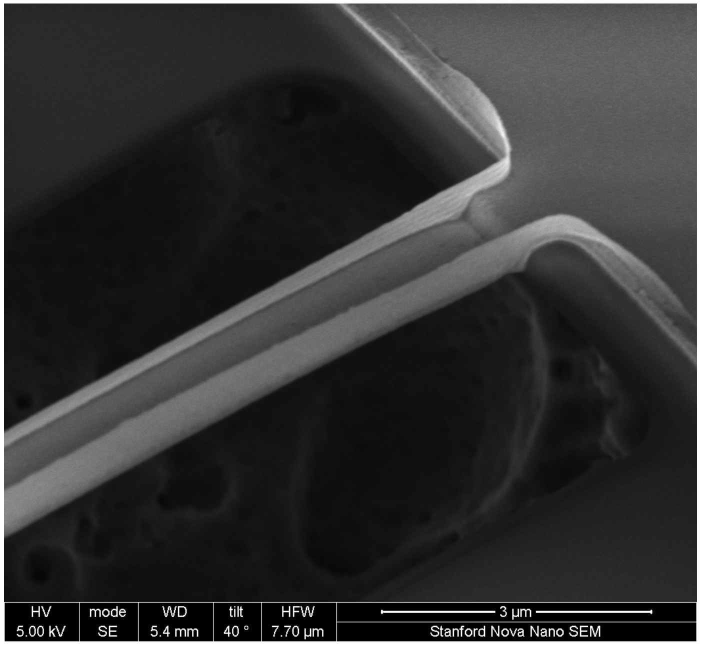
Substrate Biasing

- Plasma flickers due to brushes losing contact when substrate holder rotates during deposition -> workaround by lowering stage 2-3 turns

Platinum Etching

Ion Milling

- Standard Recipe in mrc Etch Rate: 8.3 nm/min
- Redeposition on resist sidewalls
- Poor selectivity and damage to underlying layer
- Works, even for small (500 nm) features



Dilute Aqua Regia

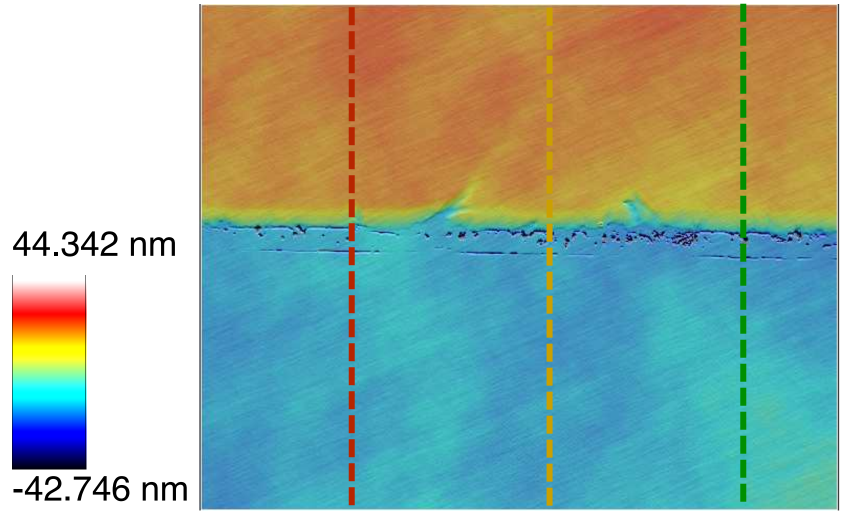
- 3:1:2 mixture of $HCl:HNO_3:H_2O$
- Self-heats to 34 C
- Literature Etch Rate: 3.5 nm/min
- Excellent selectivity towards SiO_2
- Does not etch Pt
- … unless pre-treating surface with Ar plasma or 50:1 HF dip
- Doesn't work for small (5 um) features due to excessive undercut

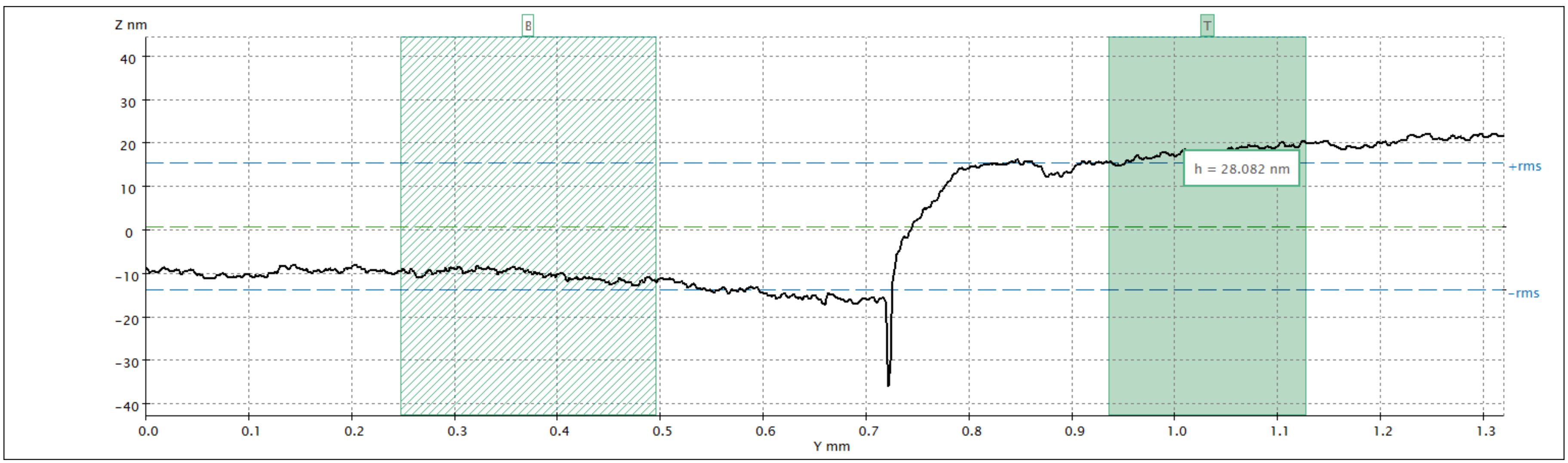
s-neox Step Height

- 25% Variation for thin (~30 nm) films!

	dL µm	dz nm	a °	h nm
1	642.42	31.590	0.0028	33.136
2	639.84	33.367	0.0030	35.208
3	651.45	26.142	0.0023	28.082
4				
5				
Avg	644.57	30.366	0.0027	32.142
Std	4.9776	3.0741	0.0003	2.9929

	h [nm]
1	33.136
2	35.208
3	28.082





Cursor: dz = 26.142 nm dL = 651.45 µm

Thickness Measurement on Thin Metal Films

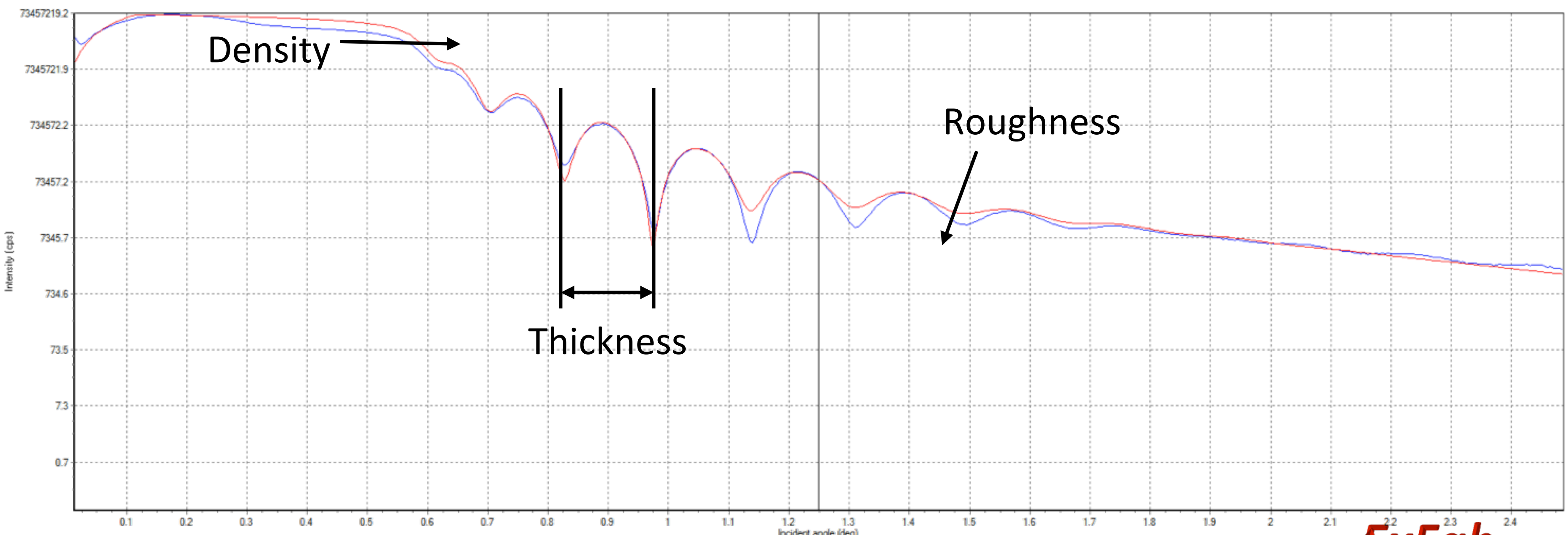
s-neox	alphastep	AFM	XRR	Filmetrics	woollam
+ fast, cheap	+ fast, cheap	- slow	- slow, pricey	+ fast, cheap	+ fast
+ non-contact	- sample contact	- sample contact	+ non-contact	+ non-contact	+ non-contact
- low temp only, needs Kapton tape	- low temp only, needs Kapton tape	- low temp only, needs Kapton tape	+ high temp possible, no Kapton tape	+ high temp possible, no Kapton tape	+ high temp possible, no Kapton tape
- unreliable below 100 nm	- unreliable below 30 nm	- always unreliable	- unreliable below 5 and above 100 nm	- fails above 12 nm	- fails above 12 nm
			- affected by roughness		+ higher accuracy vs. Filmetrics

best in most cases (XRR)

best below 10 nm (woollam)

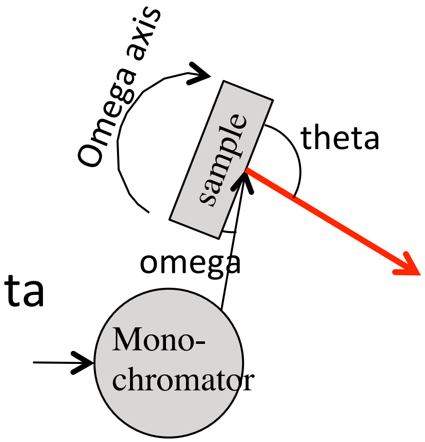
X-Ray Reflectivity (XRR)

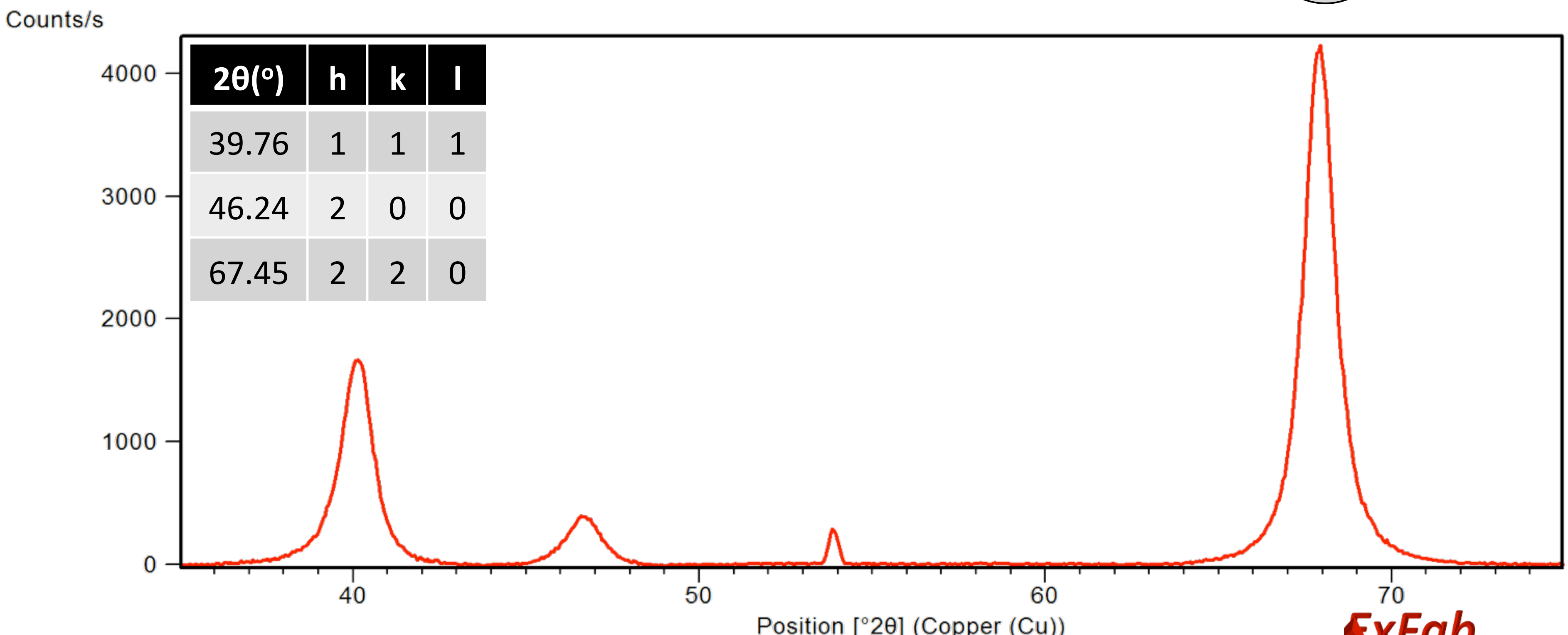
- Can be used to determine the density, thickness, and roughness of film
- Limitations:
    1. Roughness of the film
    2. Fails to Fit or Inaccurate for Films thinner than ~3 nm
    3. Unreliable Density Fitting – usually best to fix at bulk value



X-Ray Diffraction (XRD)

- Non-symmetric Scan (Glazing Incident Scan / 2-theta)
  - Gives information about the crystallinity of Pt film
  - Does not give meaningful data FWHM / peak height data

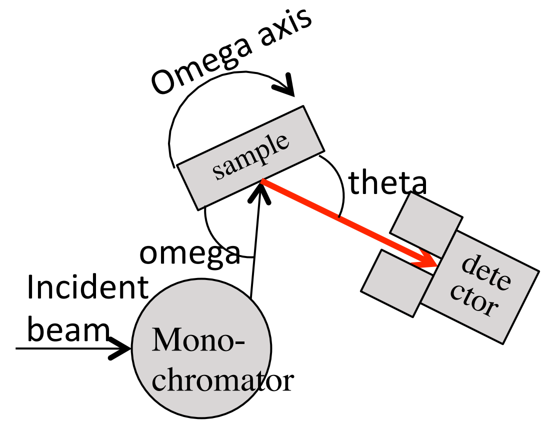


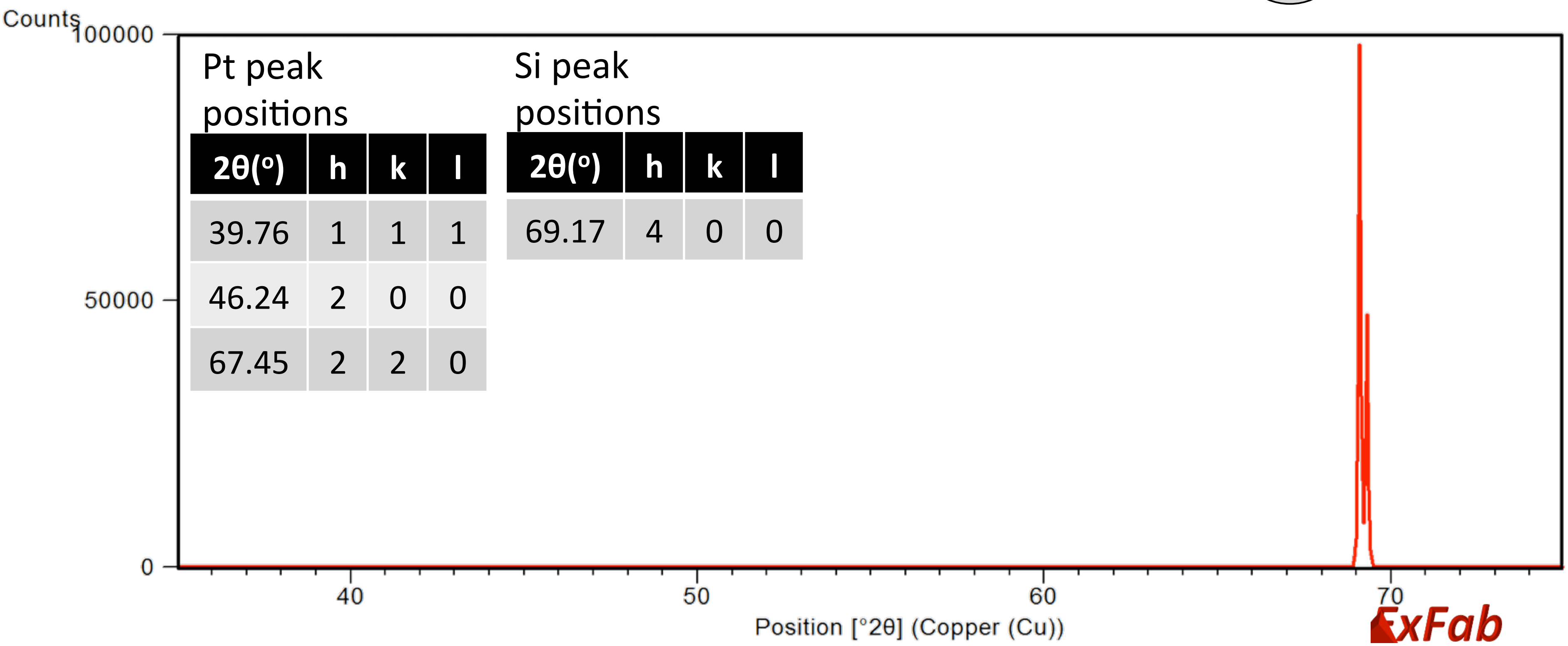


2θ(°)	h	k	l
39.76	1	1	1
46.24	2	0	0
67.45	2	2	0

X-Ray Diffraction (XRD)

- Symmetric Scan (Theta-2Theta measurement)
  - Provides meaningful data (e.g. grain size, texture)
  - Si (400) overlaps with Pt (111)





Pt peak positions

2θ(°)	h	k	l
39.76	1	1	1
46.24	2	0	0
67.45	2	2	0

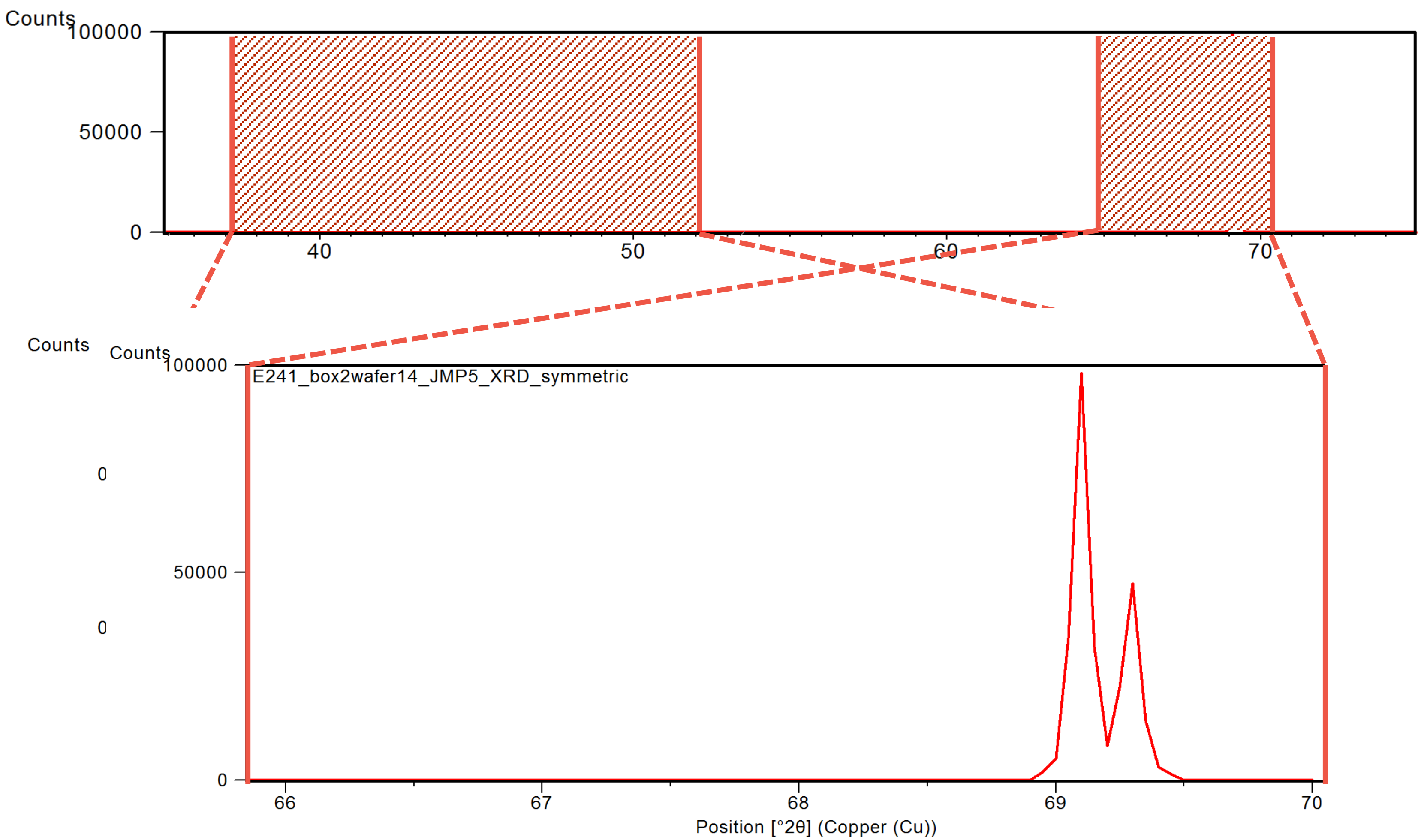
Si peak positions

2θ(°)	h	k	l
69.17	4	0	0

Image from http://prism.mit.edu/xray

X-ray Diffraction (XRD)

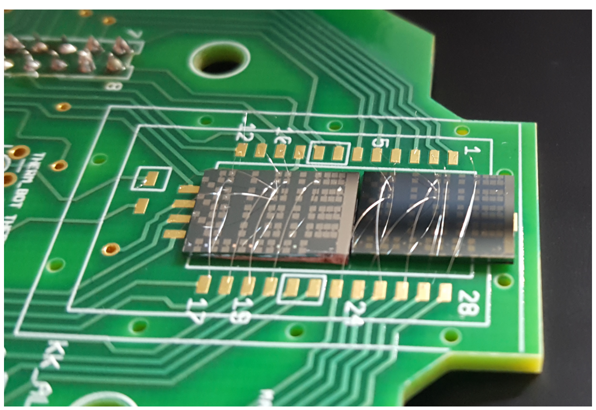
Use Si(111) !



Electrical Measurements

Room-Temperature Resistivity

- cascade
  - IV measurement with Keithley 4200 on 4-wire structures
  - Errors from wider Pt beams by Litho and redeposition
- prometrix
  - 4-probe testing on unpatterned film
  - Very consistent – average StdDev 0.5%
- cascade and prometrix within 1%



Temperature-Controlled Setup

- Wirebonding - can successfully bond to 30 nm sputtered Platinum
- Stability tests:
  - Devices stabilized in oven at 30 °C
  - Resistance measured ~ every 1 sec for 30 min
- TCR measurements:
  - Stabilize oven at temperatures from 10 °C to 50 °C
  - Measure resistance at each temperature
  - Fairly time consuming because of oven stabilization time

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Thank You.