

# Testing the Limits of Designed Experiments

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18 July 2012

# The Value of Replication in Experimentation



- A single good result may look nice in your thesis, but.....
  - Was it a lucky coincidence that you can never repeat?
  - Will other labs confirm the result, or will they prove that it's misleading?
  - Will it have any impact on high-volume manufacturing?
- Some rules for estimating whether a result is “real” or just a fluke:
  - It takes three *independent* replicates to give a trustworthy average
  - It takes ten *independent* replicates to give a trustworthy standard deviation
- *Independent* data capture the sources of uncontrollable variation
  - Cell-to-cell
  - Wafer-to-wafer
  - Day-to-day
  - Tool-to-tool? Person-to-person? Lab-to-lab?
- Example: 8 small cells on each of 10 wafers processed in 2 lots of 5
  - For multi-Si, typically every cell is independent (80 replicates)
  - For mono-Si, probably only every lot is independent (2 replicates!)

# Statistical Significance: A-B Comparison



- Common definition of “statistically significant difference”:
  - There is less than a 5% chance that the observed difference between the two things being compared is just due to random noise (p value).
  - If  $p < 0.05$ , the better process will likely be the better process in the long run.
  - If  $p > 0.05$ , you may need more data, or the difference may not be important.
- Conclusions that are based on results that are not statistically significant:
  - Usually lead to a great deal of wasted time and effort!
  - Commonly occur when the first result is the desired result.
  - People like to see what they expect. People don’t like repetition.

## One-way ANOVA: Reflectance(%) versus Process (3 runs each)

Source	DF	SS	MS	F	P
Process	1	5.134	5.134	10.00	0.034
Error	4	2.053	0.513		
Total	5	7.187			

Mean of each process lies beyond the 95% confidence limit for the mean of the other process -> Stat Sig!

S = 0.7164    R-Sq = 71.43%    R-Sq(adj) = 64.29%

Individual 95% CIs For Mean Based on Pooled StDev

Level	N	Mean	StDev	CI Lower	CI Upper
New	3	13.307	0.374	(-----*-----)	(-----+-----)
Old	3	15.157	0.942	(-----*-----)	(-----+-----)

# D-I-Y 95% Confidence Intervals



df	$t_{.025}$	df	$t_{.025}$	df	$t_{.025}$	df	$t_{.025}$
1	12.71	6	2.45	12	2.18	25	2.06
2	4.30	7	2.36	14	2.14	30	2.04
3	3.18	8	2.31	16	2.12	40	2.02
4	2.78	9	2.26	18	2.10	60	2.00
5	2.57	10	2.23	20	2.09	120	1.98

With 95% confidence, the *true mean* lies within the range given by this expression

$$\longrightarrow m \pm \frac{t_{.025} S_p}{\sqrt{n}}$$

Where:

m is the measured average (mean) value of the data

$t_{.025}$  is from the above table (Student's-t)

$S_p$  is the pooled standard deviation of the available data\*

n is the number of data points averaged to obtain m

For A-B comparison example:  
 df = 4\*\*, so  $t_{.025}=2.78$   
 $S_{old} = .94, S_{new} = .37, S_p = .72$   
 $n_{old}=3, n_{new}=3$   
 $m_{old}=15.2, m_{new}=13.3$

\*Can be based on more data than just what was used to calculate m (if all data should have same Stdev)

\*\*df = total number of data points used minus the number of different mean values represented

# Choosing Experimental Factors



- Think of all of the factors that might affect your result
  - It's better to be all-inclusive to avoid overlooking something important
  - Decide which factors are readily controllable and which are not
  - Rank controllable factors by significance (performance, cost, repeatability)
  - Design experiment to uniformly and randomly sample uncontrollable factors
- Pick several (e.g. four to six) of the most significant controllable factors
  - For each factor, pick a nominal *midpoint* value (often the current best practice)
  - Pick a range for each, within which you are confident the process won't fail
  - Adjust the midpoint to either the arithmetic mean or the geometric mean
- Example: Alkaline Texturing
  - Controllable and important: KOH concentration, Time, Temperature
  - Controllable but less important: Additive concentration, Number of wafers
  - Uncontrollable: Silicate concentration, Wafer orientation, Surface roughness
- Screen the factors to find the most important ones
  - Vary one factor at a time – OK on limited budget, but not optimal
  - Full factorial (all high/low combinations) – Thorough, but time consuming
  - Fractional factorial (resolution IV) - Usually the most efficient approach

# Screening Experimental Factors



## ○ Designing a “resolution-IV” fractional-factorial experiment

- Tests a fraction of the corners of the multi-dimensional “cube” of factor space
- This design finds the effect of each factor, undistorted by factor interactions
- Add three replicates of the center point at beginning, middle and end
- Table below shows this design for a four-factor screening experiment
  - ✓ The range for each factor is normalized to -1, 0, +1 for the low, mid, high values

Factor A	Factor B	Factor C	Factor D
0	0	0	0
-1	1	-1	1
1	1	-1	-1
1	1	1	1
-1	1	1	-1
0	0	0	0
-1	-1	1	1
1	-1	-1	1
-1	-1	-1	-1
1	-1	1	-1
0	0	0	0

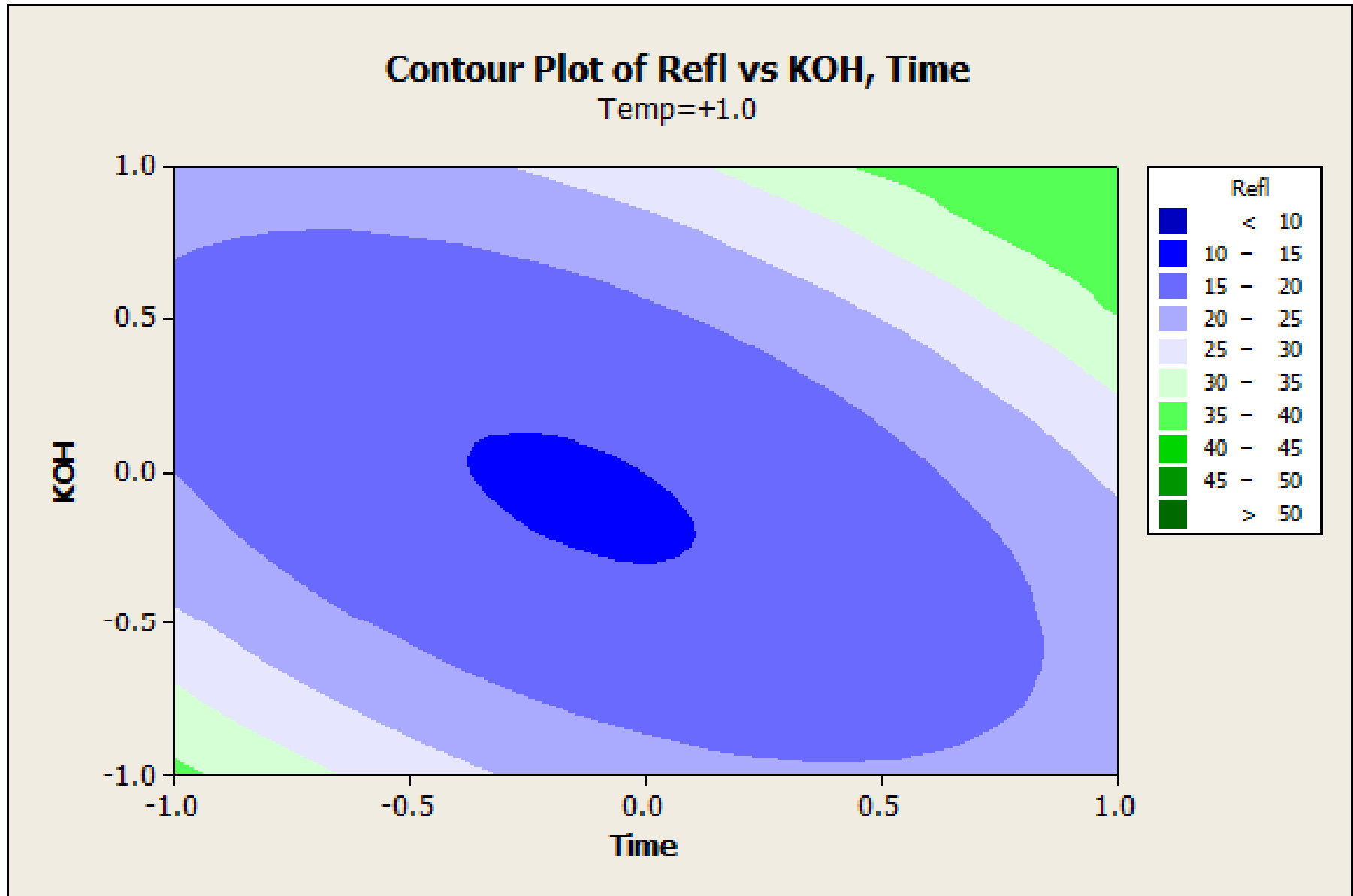
For this design, 11 runs are needed to screen these four factors to determine:

- Which factors have the most effect
- Consistency of the midpoint process
- Deviation of midpoint from linear model

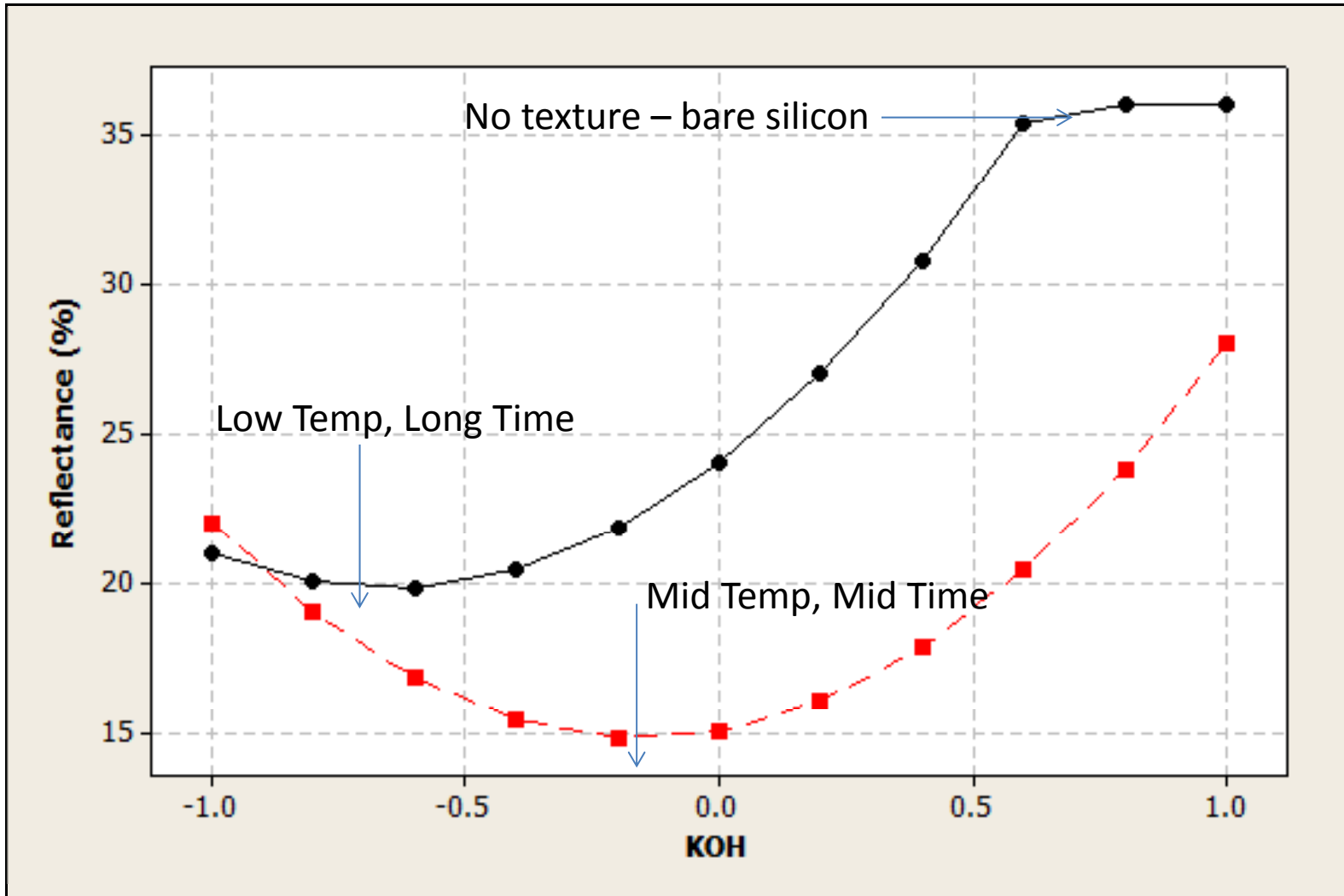


If the average at the midpoint is significantly different than the average of the corners, then expect a maximum/minimum within this space. Non-linear (squared) terms must be included in the model if you want to locate this optimum.

# Alkaline Texture Process (3 factors)



# One Factor at a Time (KOH)



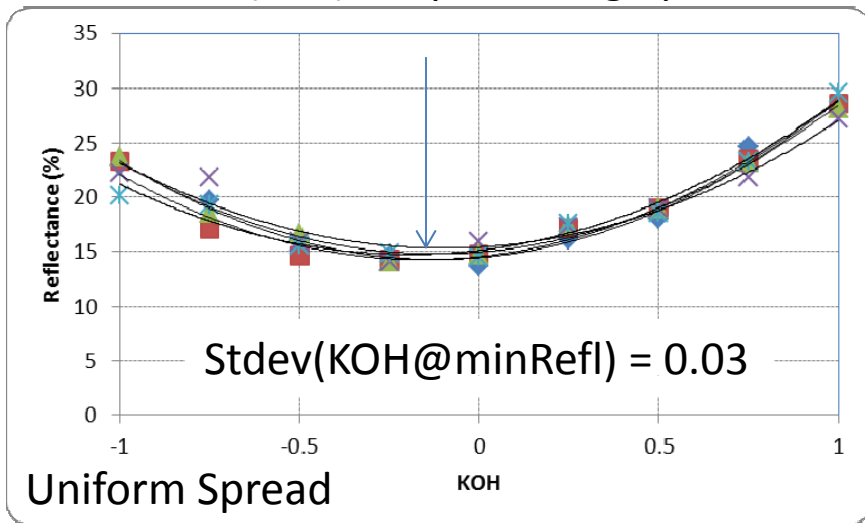
Now add experimental noise:  $y' = \text{MIN}(36, \text{MAX}(13, y + \text{StDev} * \text{NORM.INV}(\text{RAND}(), 0, 1)))$



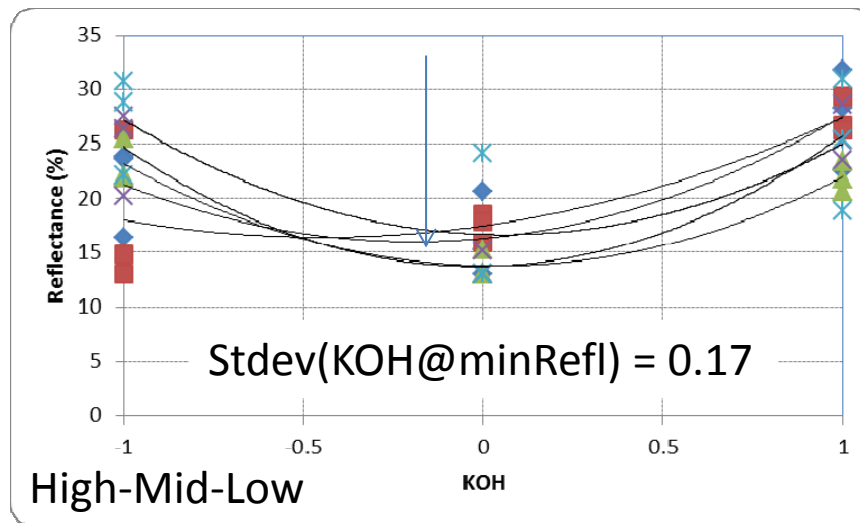
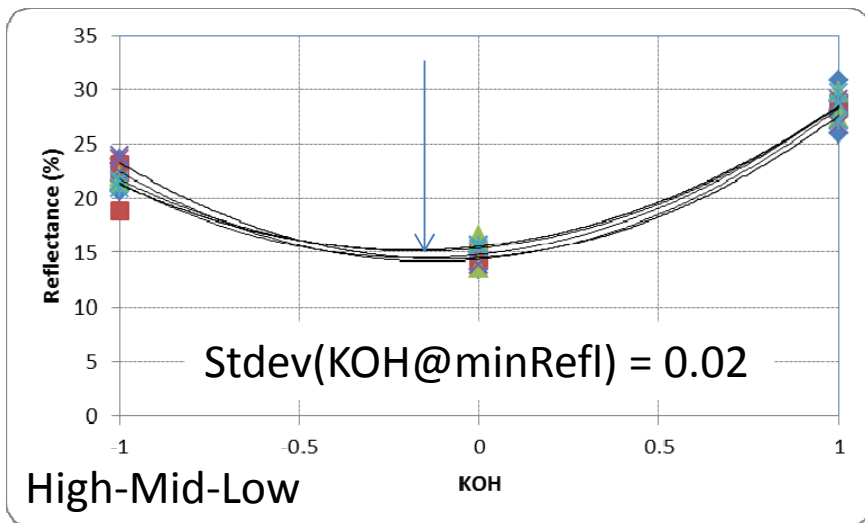
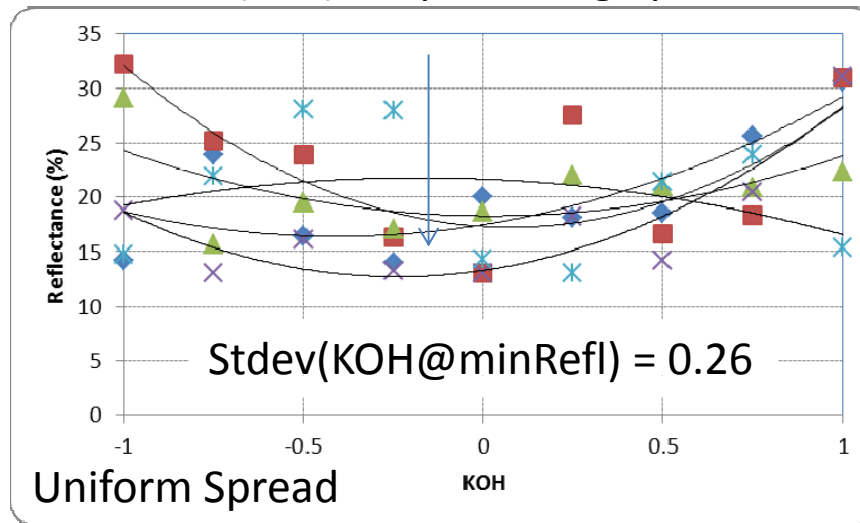
# Uniform Spread, or High-Mid-Low?



Stdev(Refl) = 1 percentage point

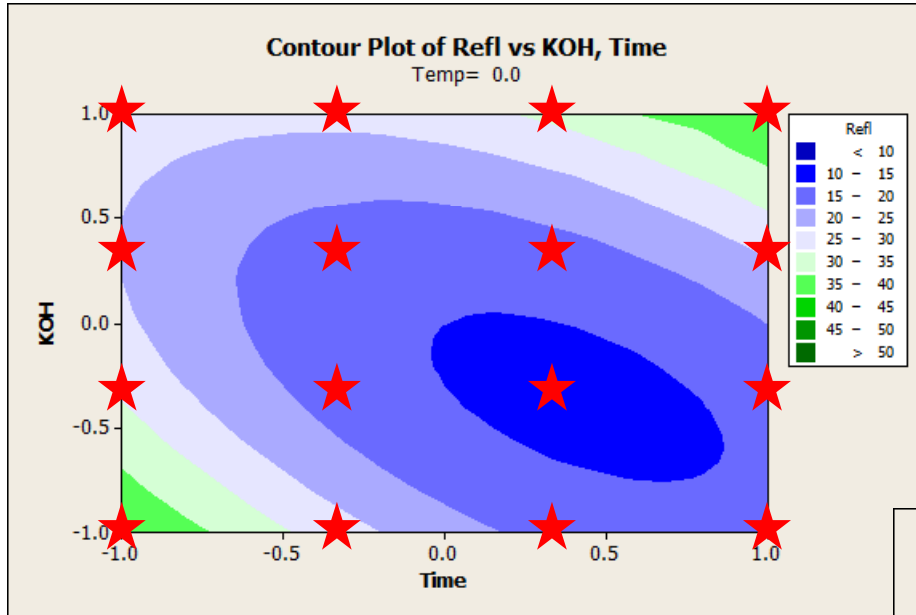


Stdev(Refl) = 5 percentage points



👉 High-Mid-Low better than Uniform Spread for normal distribution, smooth variation 9

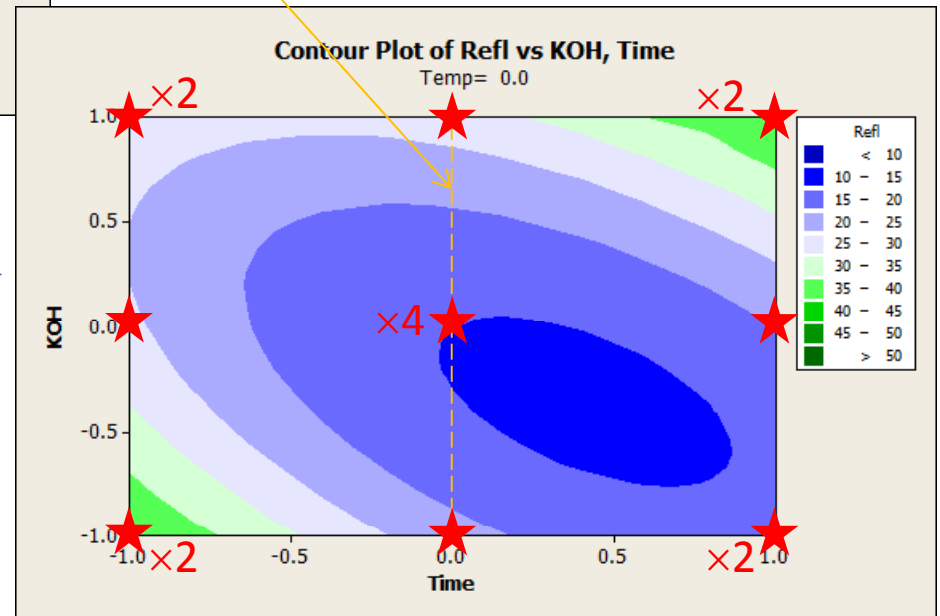
# Picking Points in Two Dimensions



← 16 points distributed uniformly:  
"Uniform Spread"

Trace of previous 1D example

16 points concentrated around perimeter and at center: "Corner-Edge-Center" →



**Note texture failure at two corners!**

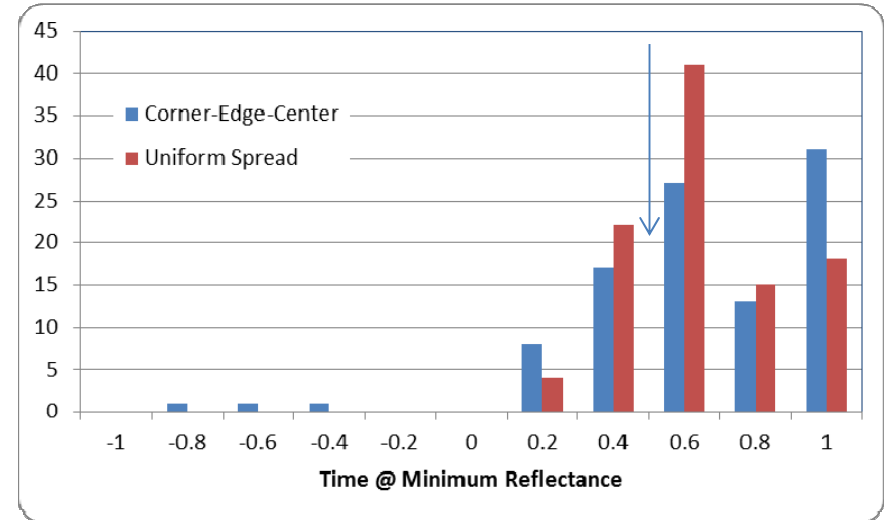
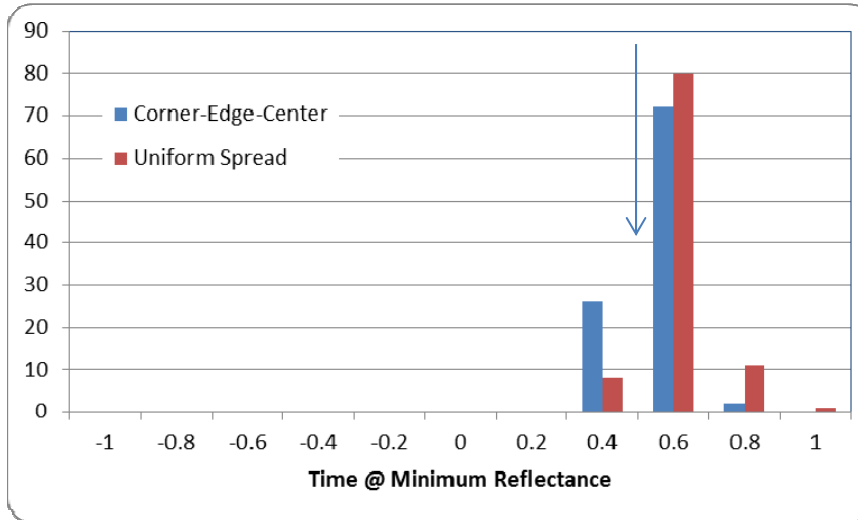
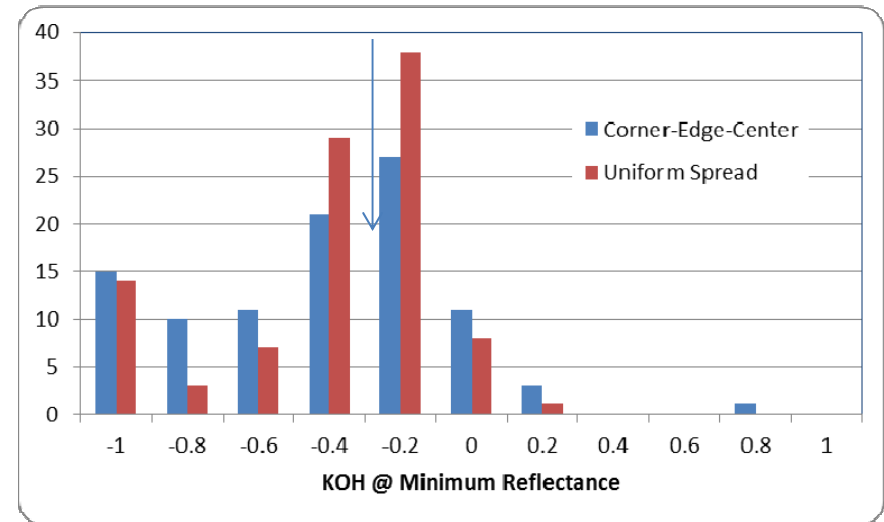
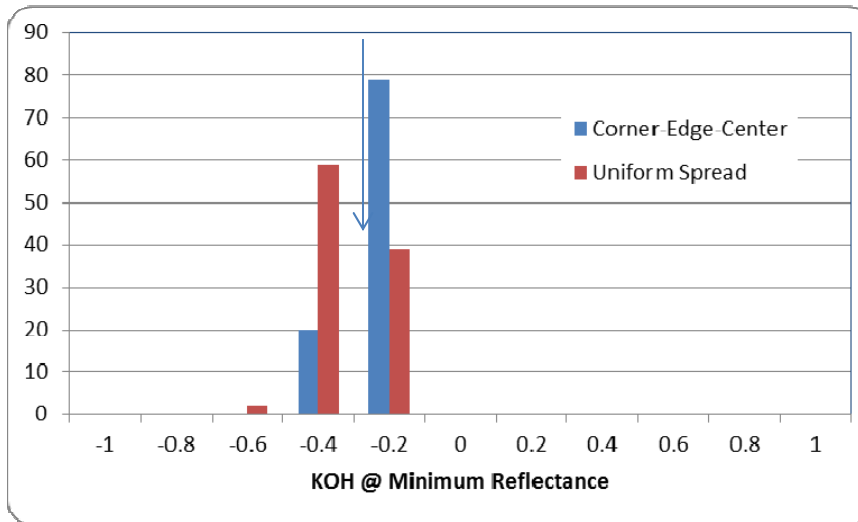
# Optimum Process from Parabolic Fit

Results from 100 16-point experiments



Stdev(Refl) = 1 percentage point

Stdev(Refl) = 5 percentage points



☞ Corner-Edge-Center still slightly better despite process failure in two corners

# 3D: Corners-Faces-Center (8+6+2=16 pts) Hanwha

## Response Surface Regression: Reflectance versus Time, Temp, KOH

The analysis was done using coded units.

Estimated Regression Coefficients for Reflectance

Term	Coef	SE Coef	T	P
Constant	16.793	1.622	10.354	0.000
Time	-2.188	1.083	-2.020	0.090
Temp	-2.534	1.083	-2.339	0.058
KOH	2.432	1.083	2.245	0.066
Time*Time	5.765	2.110	2.732	0.034
Temp*Temp	1.836	2.110	0.870	0.418
KOH*KOH	6.486	2.110	3.074	0.022
Time*Temp	1.687	1.211	1.393	0.213
Time*KOH	5.285	1.211	4.363	0.005
Temp*KOH	-1.666	1.211	-1.376	0.218

Probability that each factor could actually be zero

Maybe significant

Not significant

Statistically significant

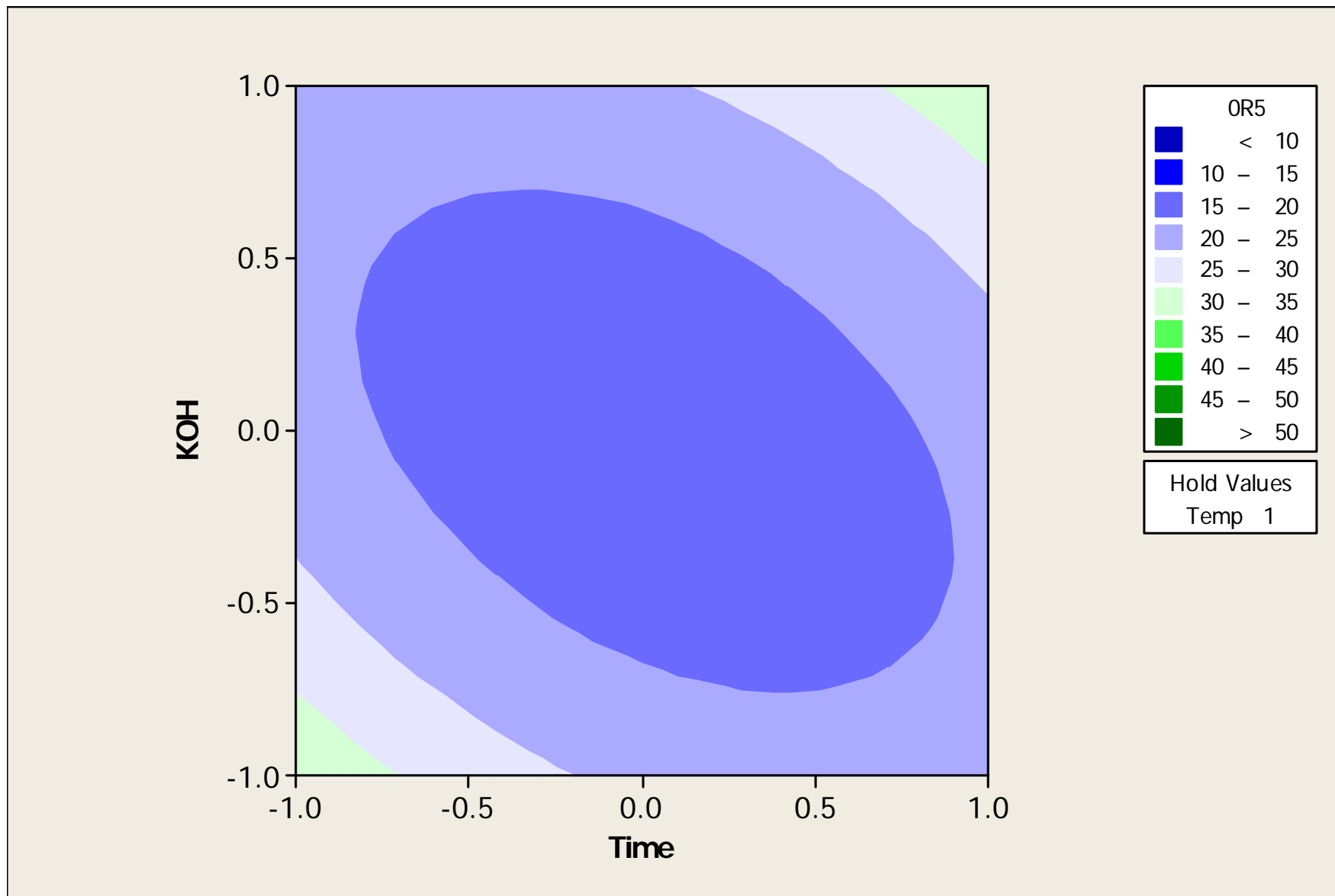
S = 3.42584 ← Stdev of the deviation of reflectance(%) from the model after accounting for the effect of the factors – due to both modeling error (3.3%) and random noise (1%).

Here, due mostly to process failure in some regions of the parameter space

# 3D Response Surface from Regression Fit



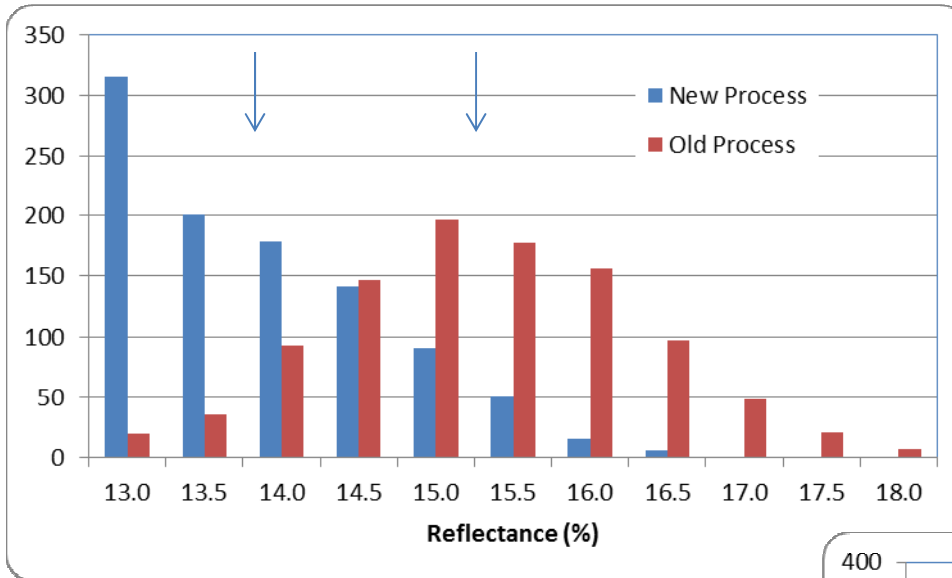
Hanwha



Old Process: Time=0, Temp=0, KOH=0

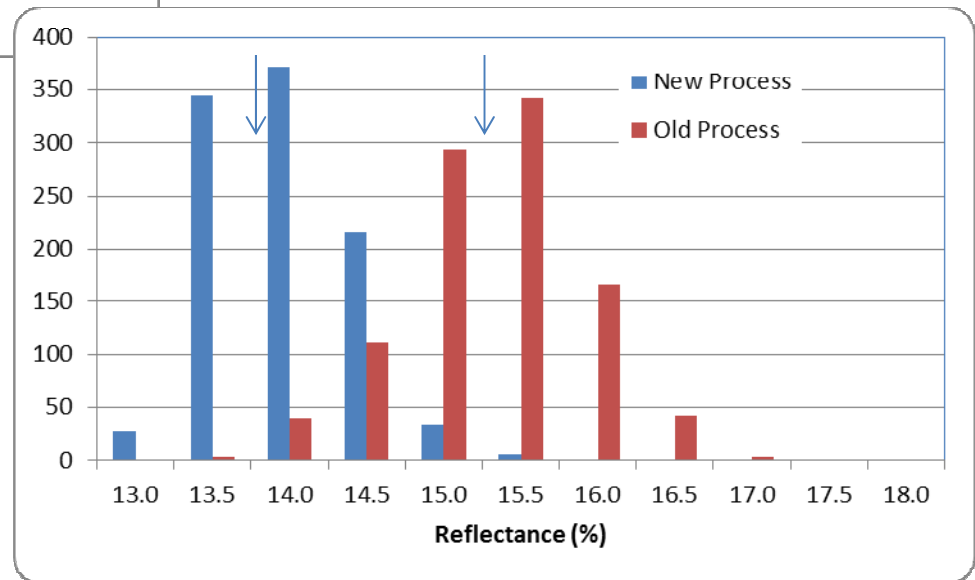
New Process: Time=0.25, Temp=0.31, KOH=-0.19

# Validation



Do one run each of the Old Process and the New Process. This is the distribution of 1000 such attempts. The new process will appear better only 86% of the time.

Compare average of three runs each of Old Process and New Process. The new process will be better 97% of the time. Note the effect of Central Limit Theorem!



The above simulations used  $Stdev(Reflectance)$  of 1 percentage point.

# Final Words of Caution

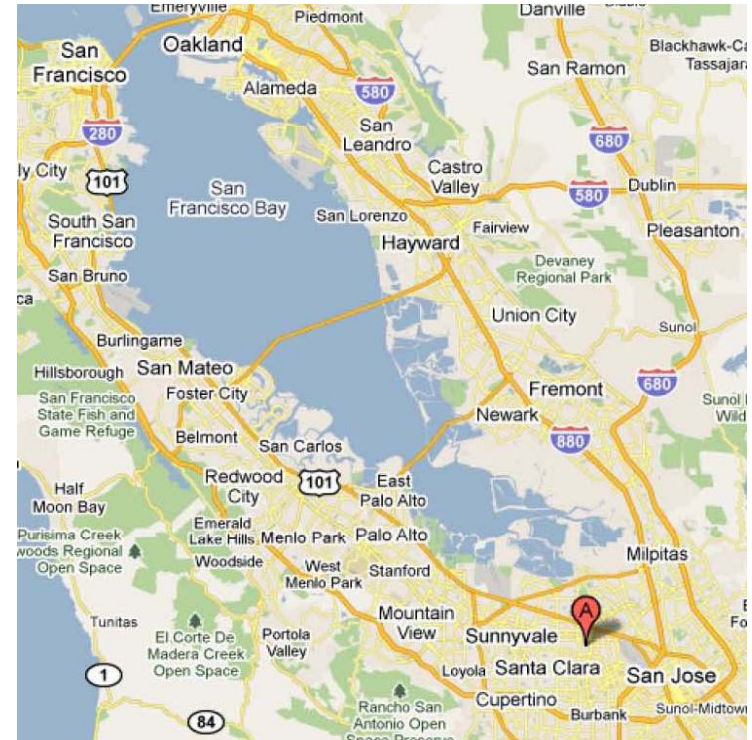


- If a result is statistically significant, still *heed the scope of the comparison*
  - If all data was collected on Monday, the conclusion may only apply on Monday
  - If all data was from a single tool, the conclusion may only apply to that tool
  - If all data used identical wafers, the conclusion may only apply to those wafers
  - If one person did all of the processing, the conclusion may only apply to them
- Rigorously keeping everything the same except the experimental factors?
  - Most likely to give a statistically significant result, *but with a narrow scope*
  - The result may not be repeatable under slightly difference circumstances!
    - ✓ Spread your replicates across multiple days
    - ✓ Randomize the run order to avoid misinterpretation of process drift over time
    - ✓ If wafer quality is the main source of variability, use an assortment of wafers
    - ✓ If you can, obtain replicates using different people, tools and process materials
- The most valuable results are those that others can replicate
  - In PV, unless it gets put into high-volume manufacturing, what's the point?
  - Collaboration between different R&D labs is a good thing!

***In publications, show statistical significance and clarify the scope of your results***



# When in the Bay Area, Visit the ARDL!



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