Design of Experiment and Machine Learning for the Optimization of Solar Cell Performance

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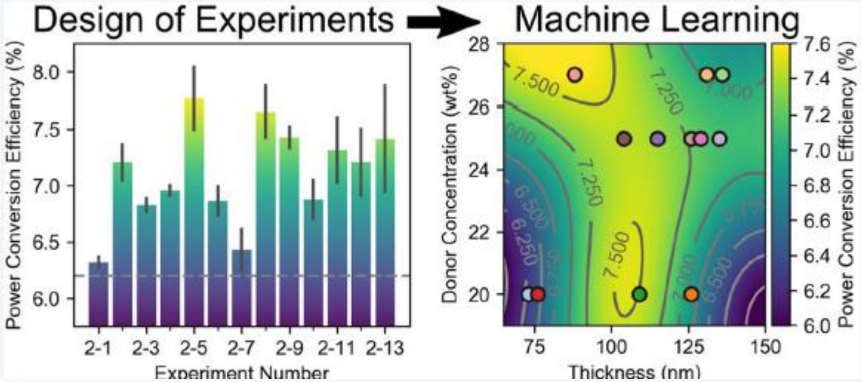
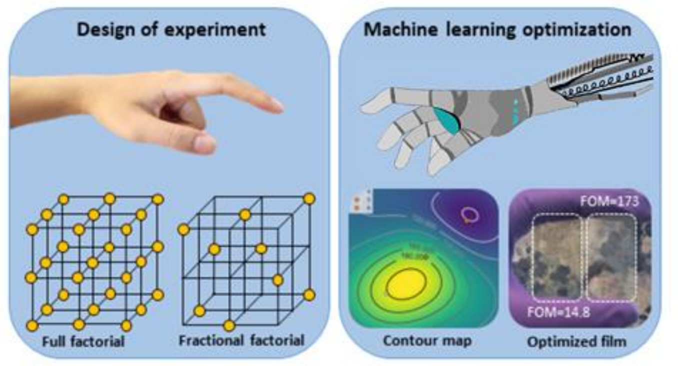




Parts of talk adapted from presentations by Lingfei Wei, UC Berkeley and Southeastern U. Nanjing

Outline

Concepts

- Design of Experiment (DOE)
- Support Vector Machine Regression
- Machine Learning and Cross-Validation

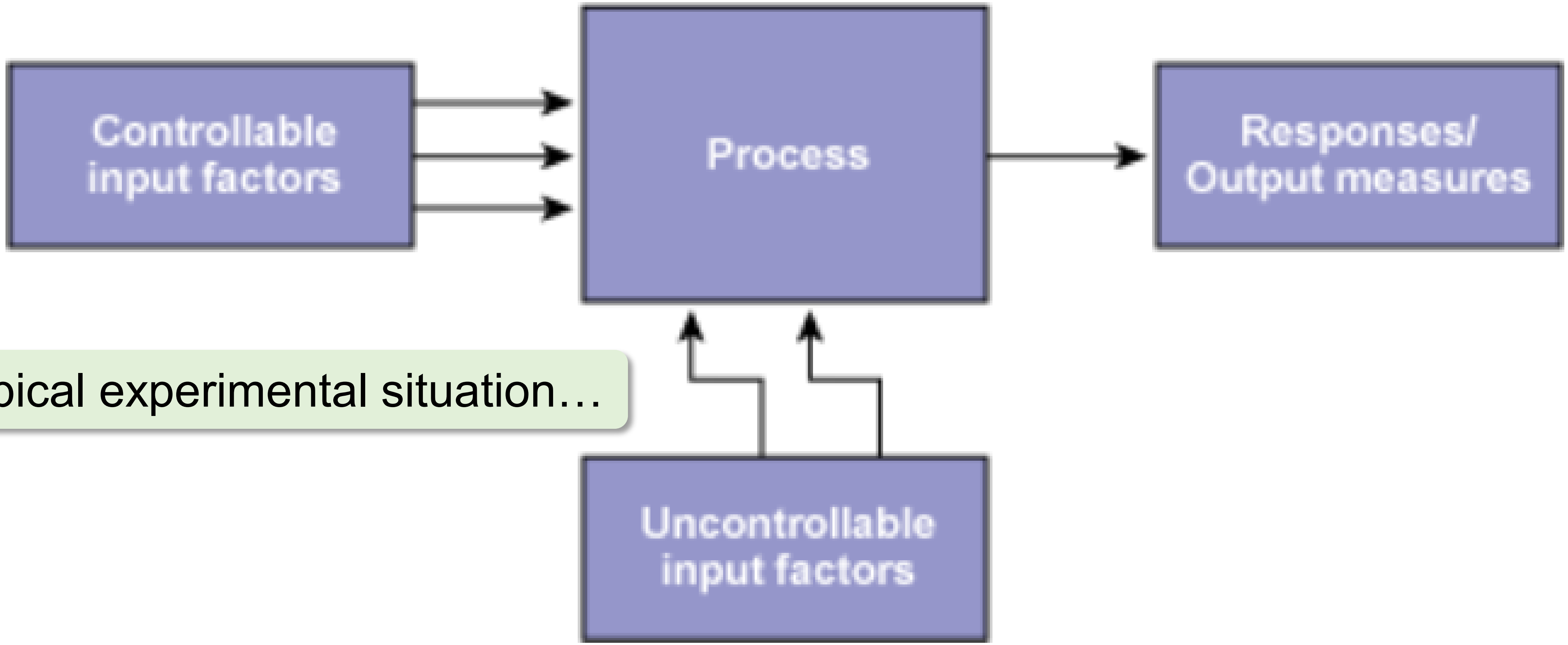
Resources

- Additional reading
- Open source software
- Commercial software

Case Studies

- Simultaneous optimization of transparency and hole conductivity in solution-deposited CuZnS
- Optimization of power conversion efficiency in solution-processed organic solar cells.

Design of Experiment

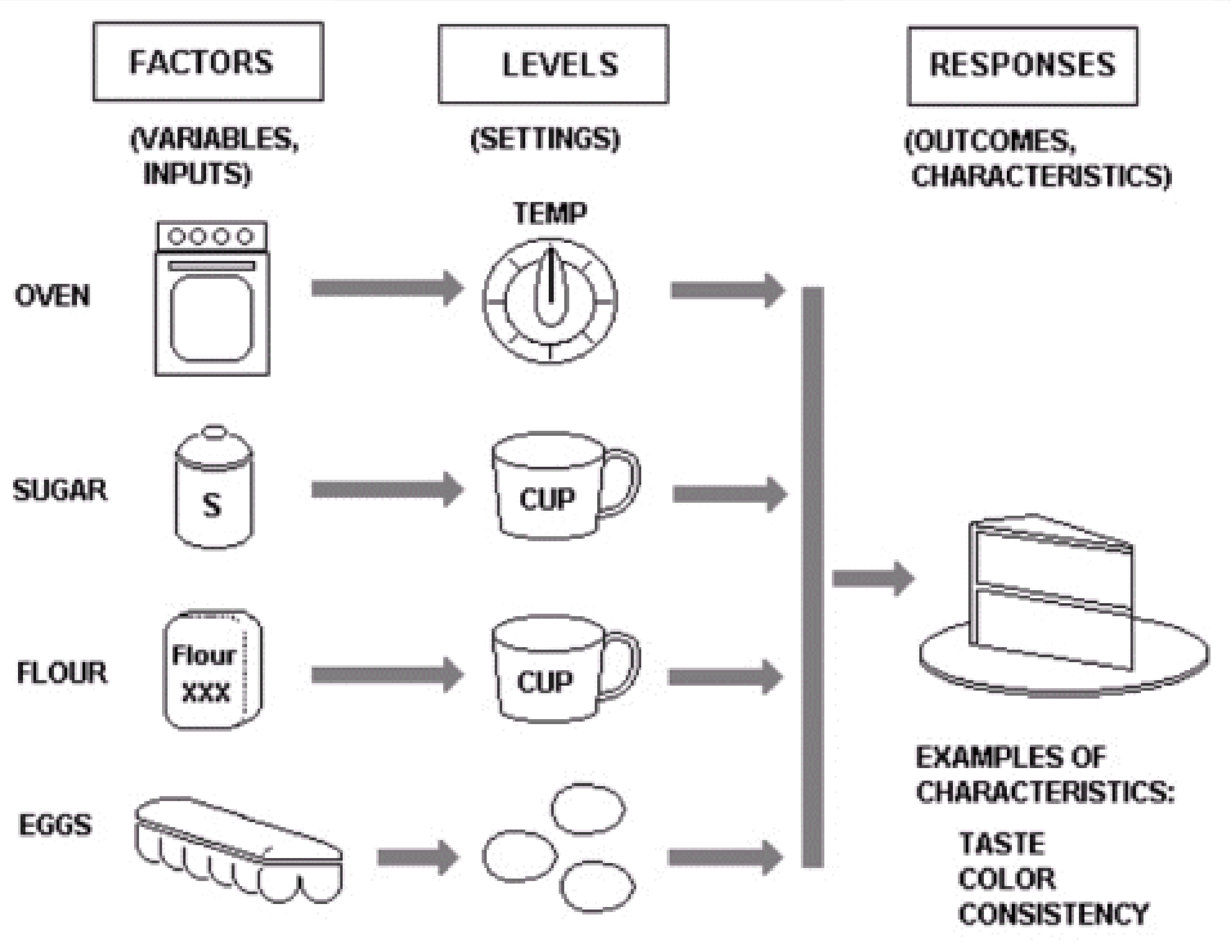


A typical experimental situation…

Design of Experiment
Goals and Nomenclature

Goals

- Achieving an optimal process output (max, min, or target)
- Comparing Alternatives
- Identifying the Significant Inputs (Factors) affecting an Output (Response)
- Reducing Variability
- Balancing Tradeoffs



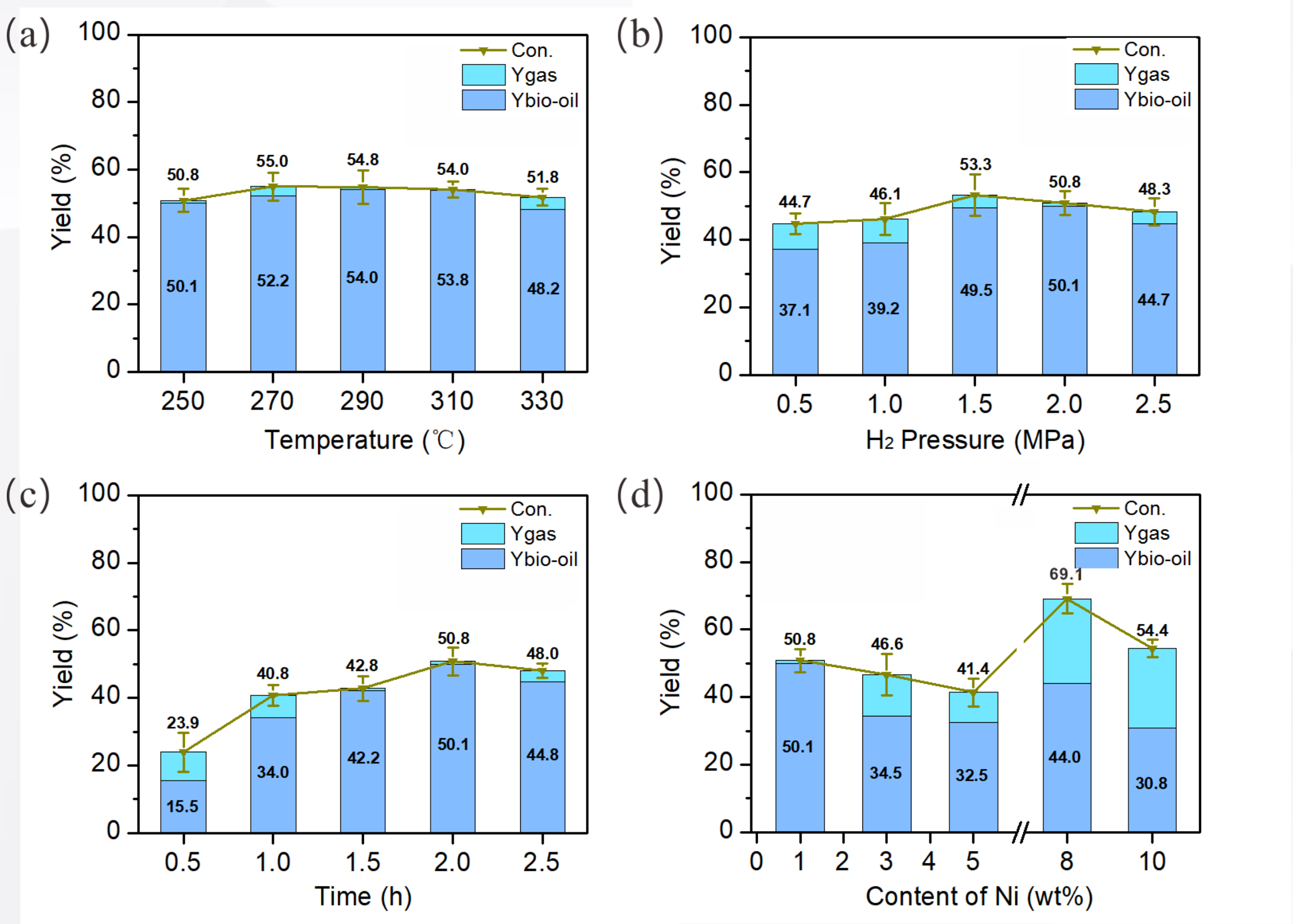
Design of Experiment

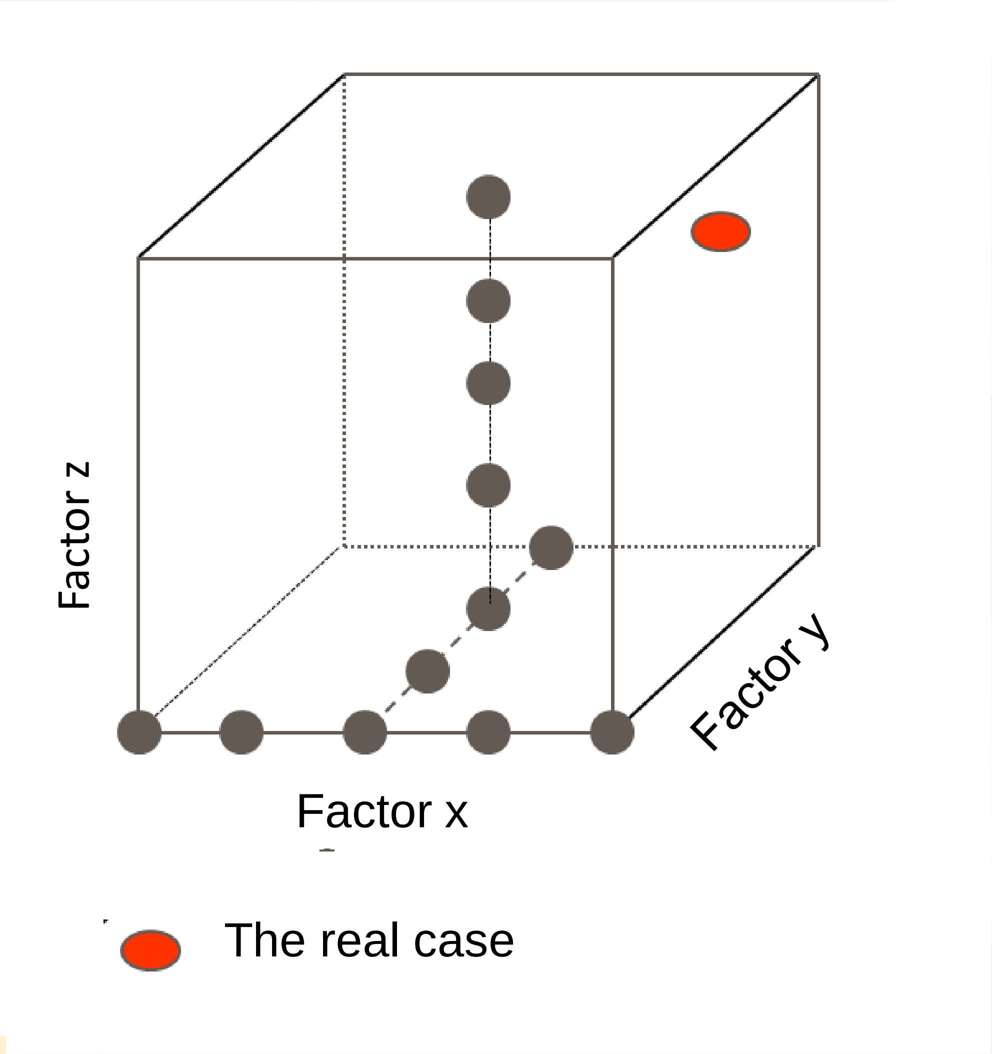
Figure 1. So many options—how do we choose? The authors pondering a small subset of the myriad of published components that have been tested in organic photovoltaic (OPV) devices. Image credit: Kelli Luber.

In this Perspective, we describe how Design of Experiments, combined with machine-learning analysis, can dramatically increase the rate of screening and optimization of materials properties and devices.

Cao, B.; Adutwum, L. A.; Oliynyk, A. O.; Luber, E. J.; Olsen, B. C.; Mar, A.; Buriak, J. M. How To Optimize Materials and Devices via Design of Experiments and Machine Learning: Demonstration Using Organic Photovoltaics. *ACS Nano* **2018**, *12*, 7434–7444.

Simple DOE: “one factor at a time”





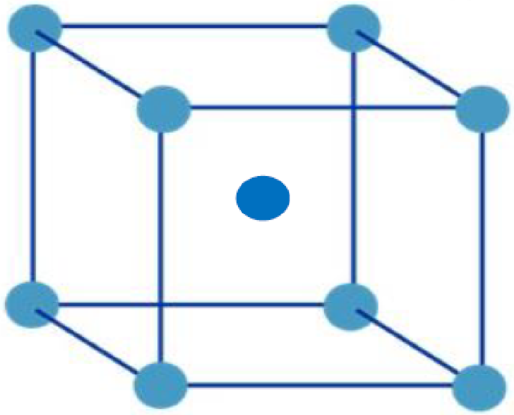
Effects of reaction temperature (a), H_2 pressure (b), reaction time (c) and content of Ni in Ni/Mo_2C catalyst (d) on the catalytic hydrogenation of lignin to bio-oil (Ybio-oil) and gas product (Ygas)

Wei, Lingfei.; Bibi, R.; Zheng, Y.; Tian, W.; Chen, L.; Li, N.; Zhou, J. *Catal. Letters* **2018**, 148, 1856–1869.

One factor at a time

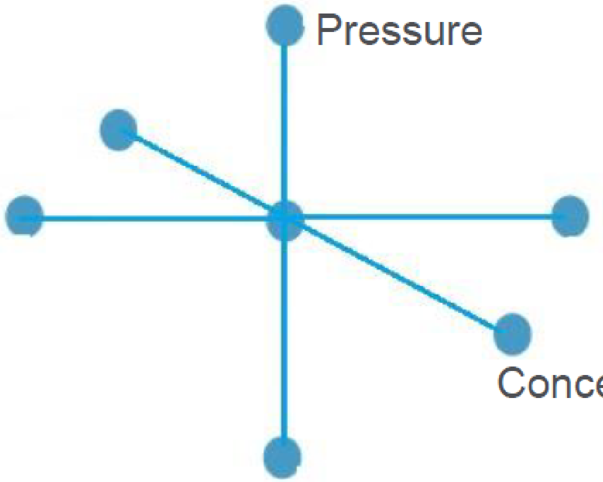
Not "wrong" but inefficient

- The Design of Experiments is the process of setting up series of tests in a efficient layout to determine what outputs result from different interaction of inputs.



Industry uses DOE to

1. Understand sensitivities
2. Capture input cross term interactions
3. The Volume of space is sampled is significantly larger than OFAT (maximum learning / wafer)



This One Factor at a time (OFAT) DOE only maps a small % of the potential process space

Chris Olsen, Sr. Director, FEP-SSG, Applied Materials, MSE-UCLA lecture, used with permission

DOE Strategy

For l levels and k factors
full factorial = l^k experiments

Number of Factors	Number of Levels per Factor	Number of Runs Full Factorial
2	2	4
2	3	9
3	2	8
3	3	27
4	2	16
4	3	81
5	2	32
5	3	243
6	2	64
6	3	729
7	2	128
7	3	2187
8	2	256
8	3	6561

How do we get to the optimal set of parameters (levels) in the minimum number of experiments?

DOE options

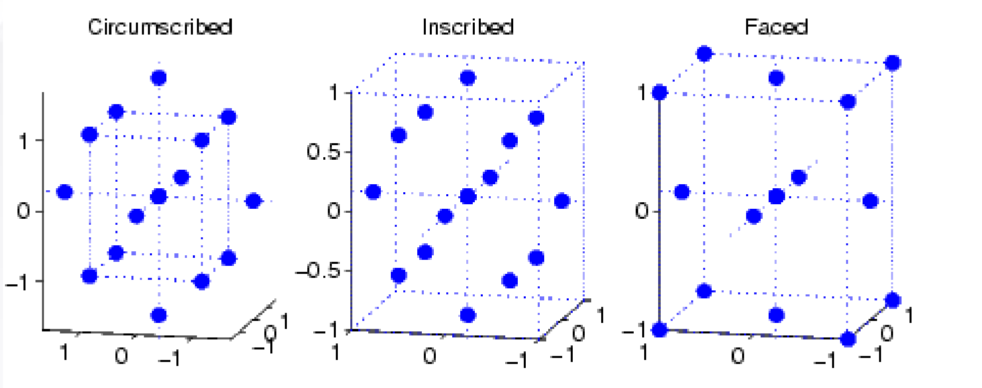
Simple case of 3 factors with 3 levels

Pressure	Temperature	FlowRate
40	290	0.2
55	320	0.3
70	350	0.4

Full factorial
$3^3 = 27$ experiments

Pressure	Temperature	FlowRa
40	290	0.2
50	290	0.2
70	290	0.2
40	320	0.2
50	320	0.2
70	320	0.2
...	...	...
...	...	...
40	290	0.3
50	290	0.3
70	290	0.3
40	320	0.3
50	320	0.3
70	320	0.3
...	...	...
...	...	...
40	320	0.4
50	320	0.4
70	320	0.4
40	350	0.4
50	350	0.4

Central-composite design
15 experiments



Pressure	Temp	Flow
55	320	0.2
55	320	0.4
55	290	0.3
55	350	0.3
40	320	0.3
70	320	0.3
Etc.		

Fractional factorial
1/3 fractional = 9 experiments

Pressure	Temp	Flow
40	290	0.2
70	290	0.3
70	320	0.2
55	320	0.3
40	350	0.3
40	320	0.4
55	350	0.2
55	290	0.4
70	350	0.4

R packages: oa.design, ccd.design

Outline

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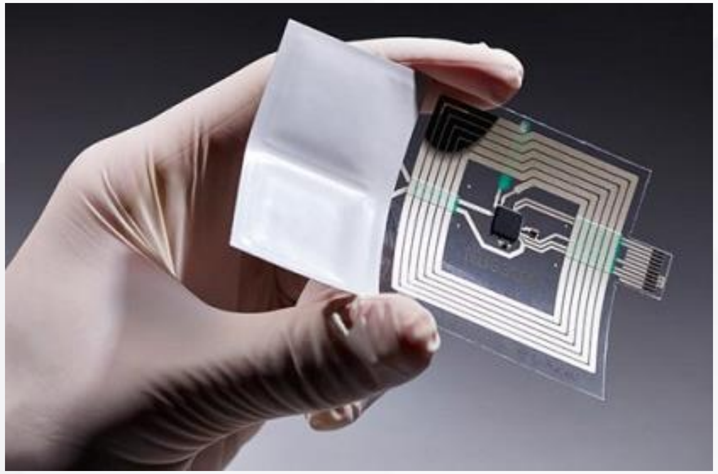
Case Studies

- Simultaneous optimization of transparency and hole conductivity in solution-deposited CuZnS
- Optimization of power conversion efficiency in solution-processed organic solar cells.

Transparent conductors

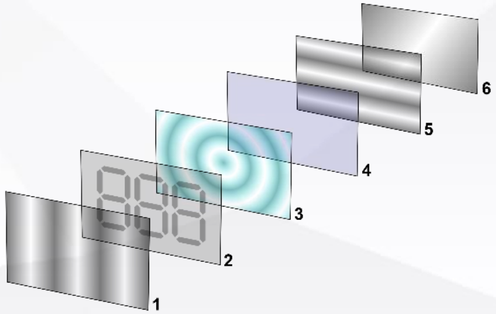


Transparent Conducting materials (TCMs)

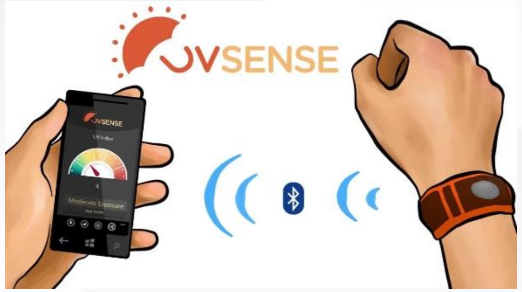
- ➢ Transparency in certain wavelength range
- ➢ Small resistivity
- ➢ Materials:
    - Metal films: Au, Ag, Cu…(t < 10 nm)
    - TCOs: ITO, IZO, AZO…
    - Conducting polymers: poly(thiophene)s (PT), Poly(fluorene)s…
    - Conductive carbon materials: CNTs, graphene

- **Applications**

Touchscreen



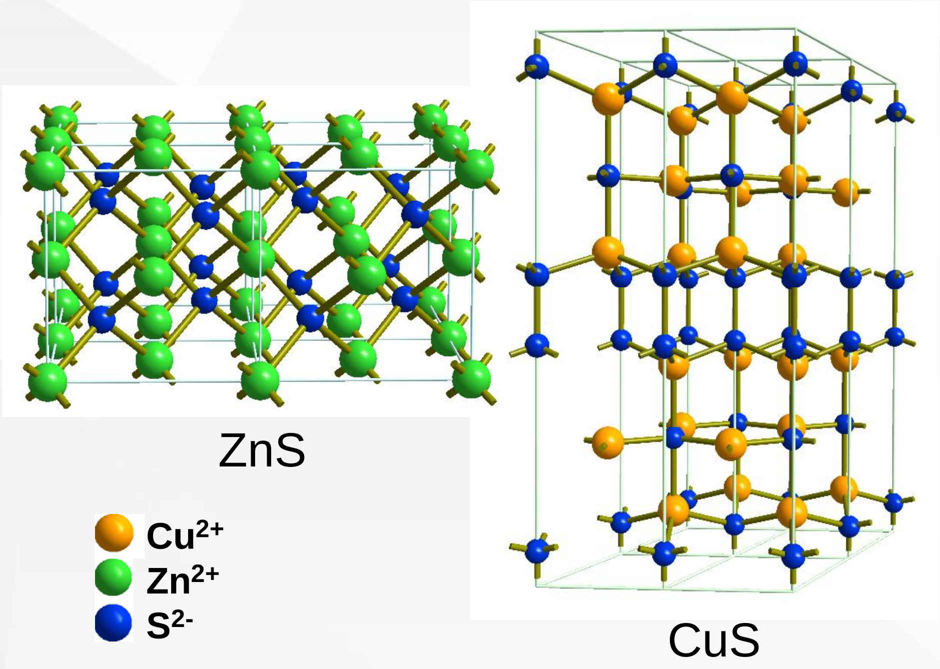
LCD layers



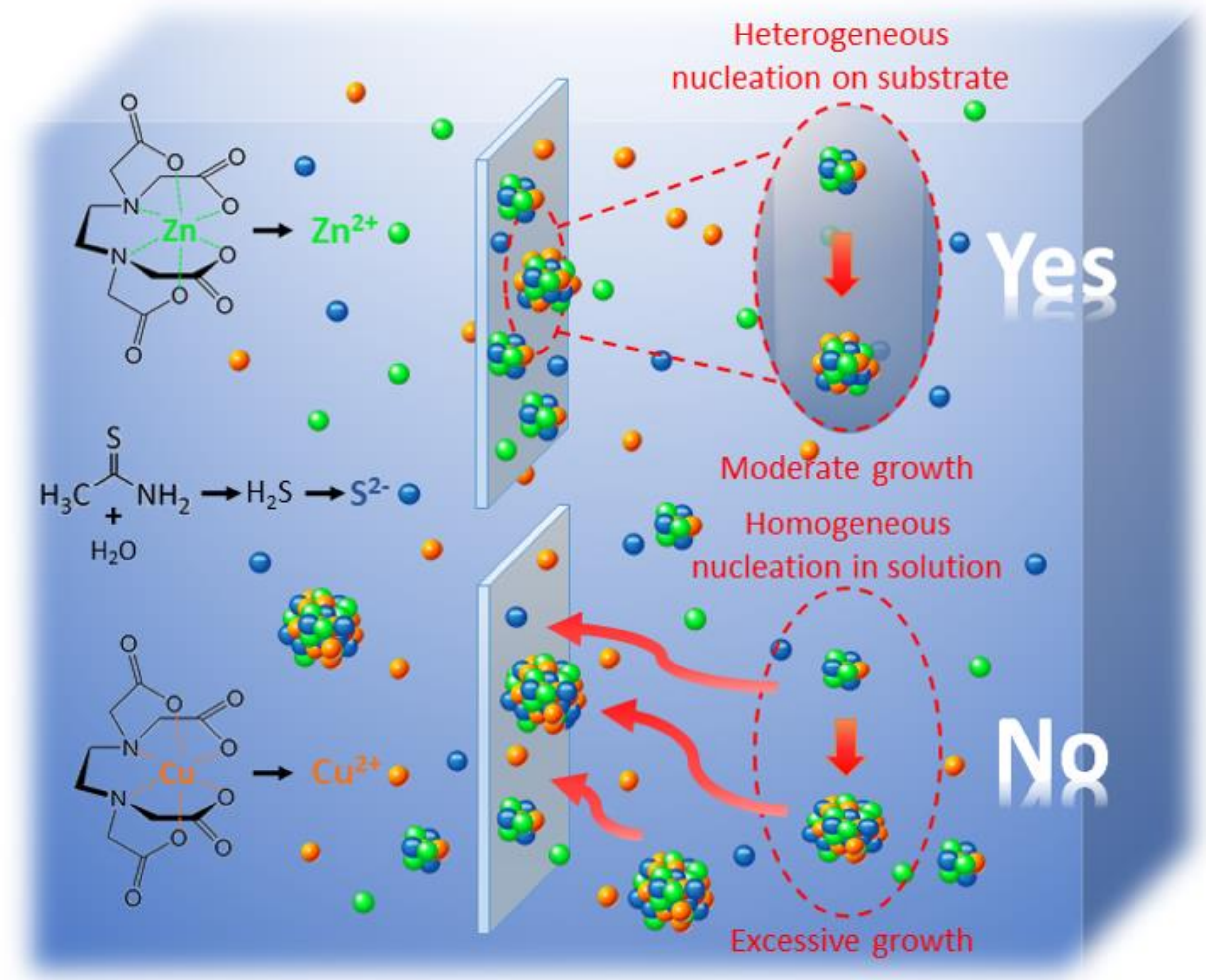
UV radiation monitor

LEDs

Photovoltaics



- Bulk CuS: p-type, E_g=~2.5 eV
- ZnS: n-type, E_g=~3.6 eV
- Thin film synthesis methods:
  - Pulsed laser deposition (PLD)
  - Sputtering
  - Electron-beam evaporation
  - Chemical bath deposition (CBD)
- $(CuS)_x(ZnS)_{1-x}$ nanocomposite:
  - hole conductivity >1000 S cm^{-1}
  - optical transmission above 70%



- Cu/Zn ratio
- Reaction temperature
- Reaction time
- Concentration of Na_2EDTA

Machine Learning

First round DOE

Factors and levels

- Cu/(Cu+Zn): 2 levels, 0.65 and 0.85
- Temperature: 4 levels, 60-90 C
- Time: 4 levels, 30-120 minutes
- Na_2EDTA concentration: 4 levels, 20-100 mM

Full factorial design = $2^1 * 4^3$ = 128 sets

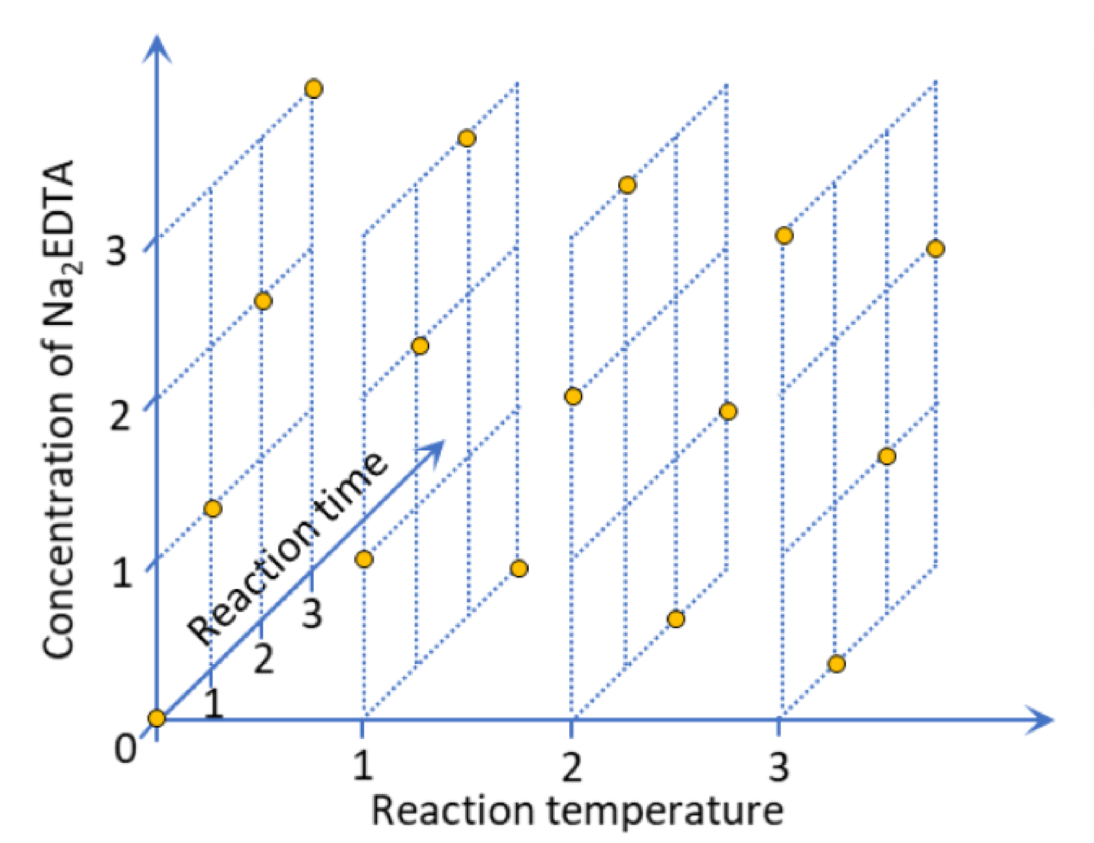
```
> oa.design(nlevels=c(4,4,4),factor.names=c('Time','Temp','EDTA'))

   Time Temp EDTA
1     3    2    1
2     3    4    2
3     2    3    4
4     2    4    1
5     2    1    3
6     4    3    1
7     1    4    3
8     4    2    3
9     1    1    1
10    2    2    2
11    3    3    3
12    4    1    2
13    3    1    4
14    1    2    4
15    4    4    4
16    1    3    2
```

Parameter range based on Xu, X.; Bullock, J.; Schelhas, L. T.; Stutz, E. Z.; Fonseca, J. J.; Hettick, M.; Pool, V. L.; Tai, K. F.; Toney, M. F.; Fang, X.; Javey, A.; Wong, L. H.; Ager, J. W. Chemical Bath Deposition of P-Type Transparent, Highly Conducting (CuS)x:(ZnS)1-x Nanocomposite Thin Films and Fabrication of Si Heterojunction Solar Cells. *Nano Lett.* **2016**, *16*, 1925–1932.

1/4 Generalized subset design

16 experiments for each Cu/(Cu+Zn) level

- Balanced: levels appear the same number of times
- Orthogonal: levels are not correlated with other



R package: oa.design

set no.	Cu/(Cu + Zn)	temperature (°C)	time (min)	concentration of Na_2EDTA (M)
1	0.65	60	30	0.02
2	0.65	60	60	0.03
3	0.65	60	90	0.07
4	0.65	60	120	0.1
5	0.65	70	30	0.03
6	0.65	70	60	0.07
7	0.65	70	90	0.1
8	0.65	70	120	0.02
9	0.65	80	30	0.07
10	0.65	80	60	0.1
11	0.65	80	90	0.02
12	0.65	80	120	0.03
13	0.65	90	30	0.1
14	0.65	90	60	0.02
15	0.65	90	90	0.03
16	0.65	90	120	0.07
17	0.85	60	30	0.02
18	0.85	60	60	0.03
19	0.85	60	90	0.07
20	0.85	60	120	0.1
21	0.85	70	30	0.03
22	0.85	70	60	0.07
23	0.85	70	90	0.1
24	0.85	70	120	0.02
25	0.85	80	30	0.07
26	0.85	80	60	0.1
27	0.85	80	90	0.02
28	0.85	80	120	0.03
29	0.85	90	30	0.1
30	0.85	90	60	0.02
31	0.85	90	90	0.03
32	0.85	90	120	0.07

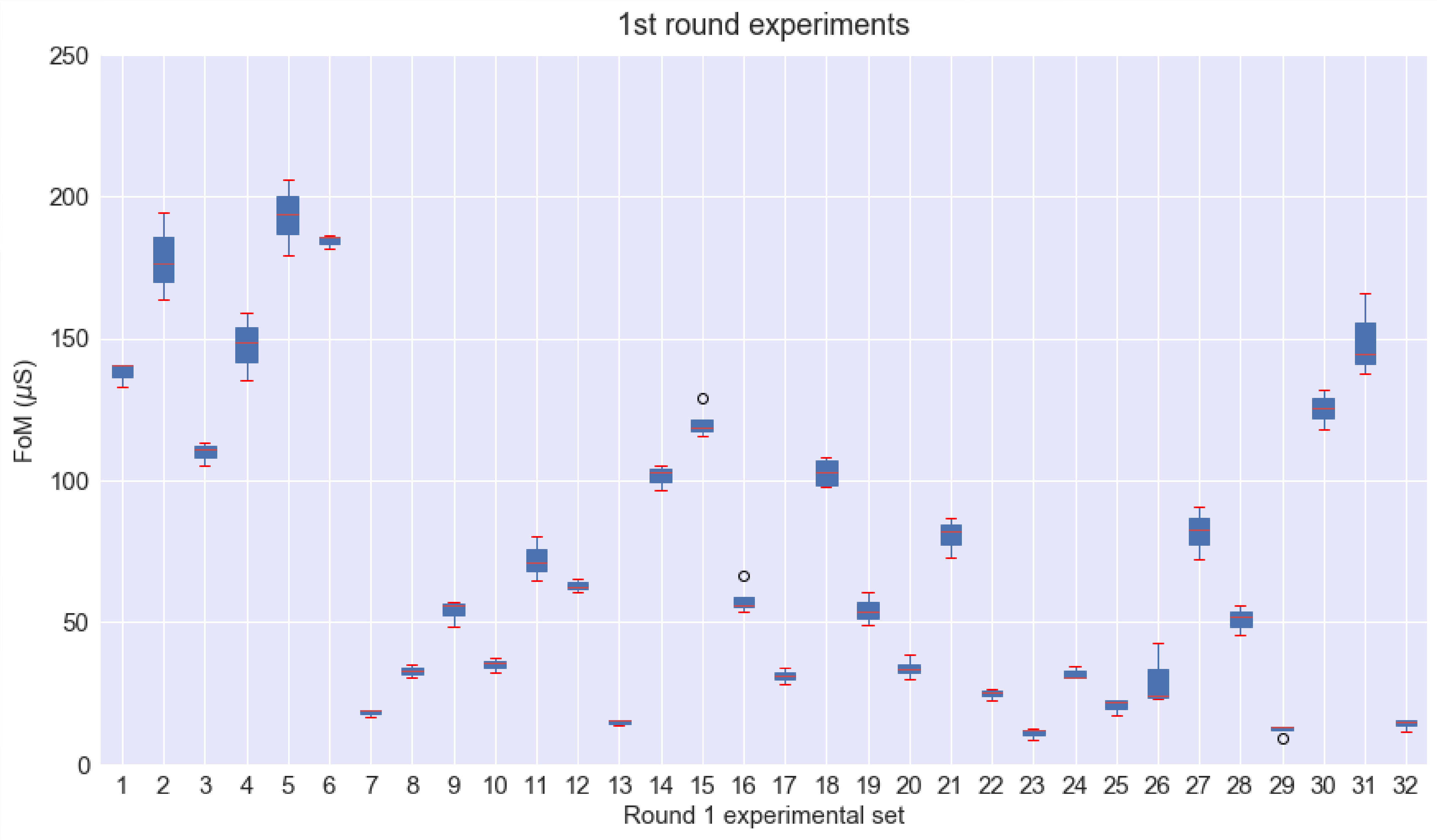
1st Run DoE

Figure of Merit (FoM, µS)

$\Phi_{TC}=T^{10}/R_s$

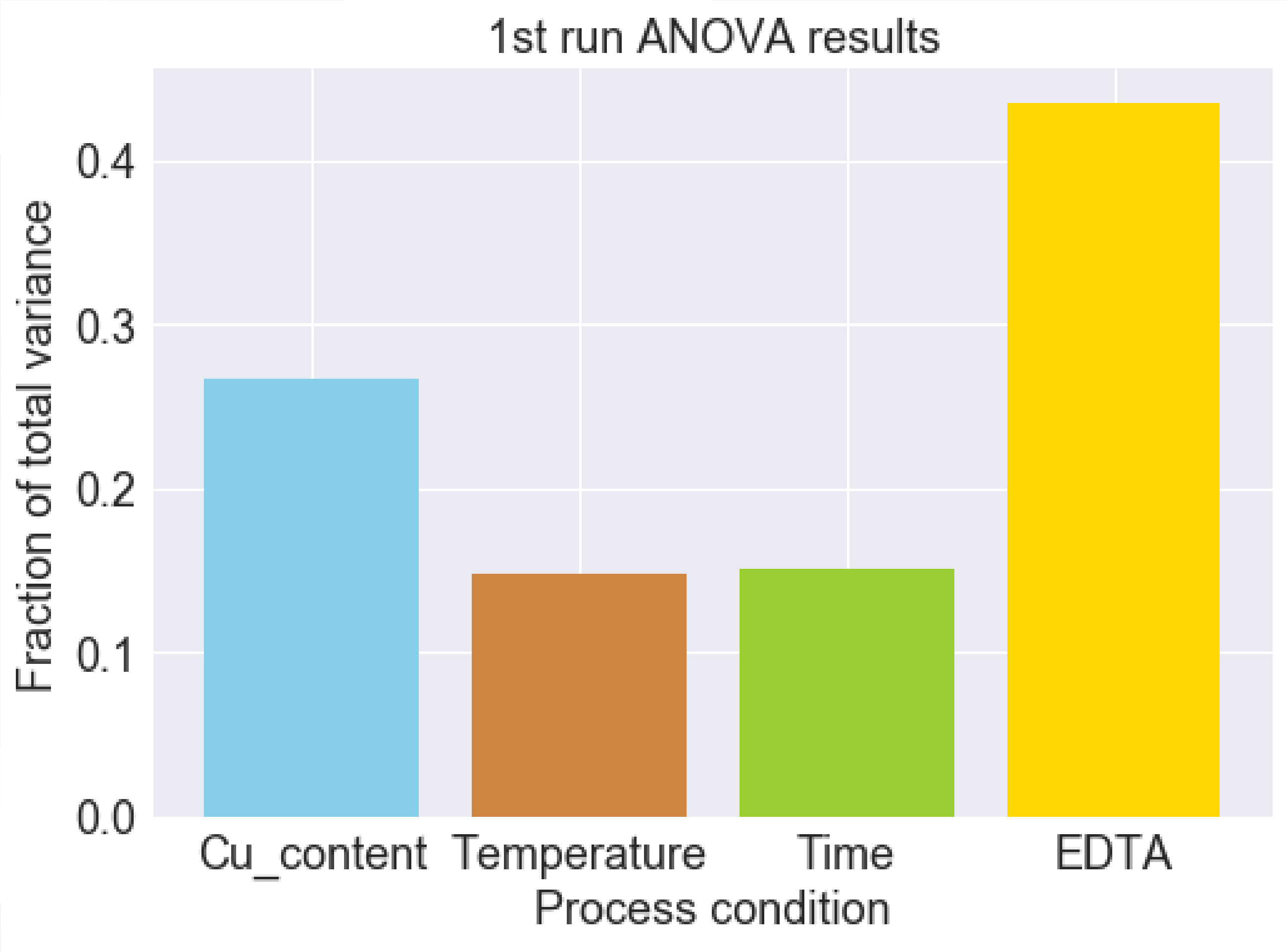
T: transmittance (380-780 nm)
R_s: sheet resistance (Ω/□)

3-4 repetitions per set, 109 total experiments



Wei, L.; Xu, X.; Gurudayal; Bullock, J.; Ager, J. W. Machine Learning Optimization of P-Type Transparent Conducting Films. *Chem. Mater.* **2019**, *31*, 7340–7350.

1st run statistics analysis (analysis of variance, ANOVA)



All 4 factors contribute to the observed variance in FoM

	df	sum_sq	mean_sq	F	PR(>F)
C(Cu_content)	1.0	48881.894831	48881.894831	37.478609	2.122641e-08
C(Temperature)	3.0	27081.122743	9027.040914	6.921191	2.924970e-04
C(Time)	3.0	27665.057097	9221.685699	7.070429	2.454998e-04
C(EDTA)	3.0	79745.058425	26581.686142	20.380647	2.946339e-10
Residual	94.0	122600.550196	1304.261172	NaN	NaN

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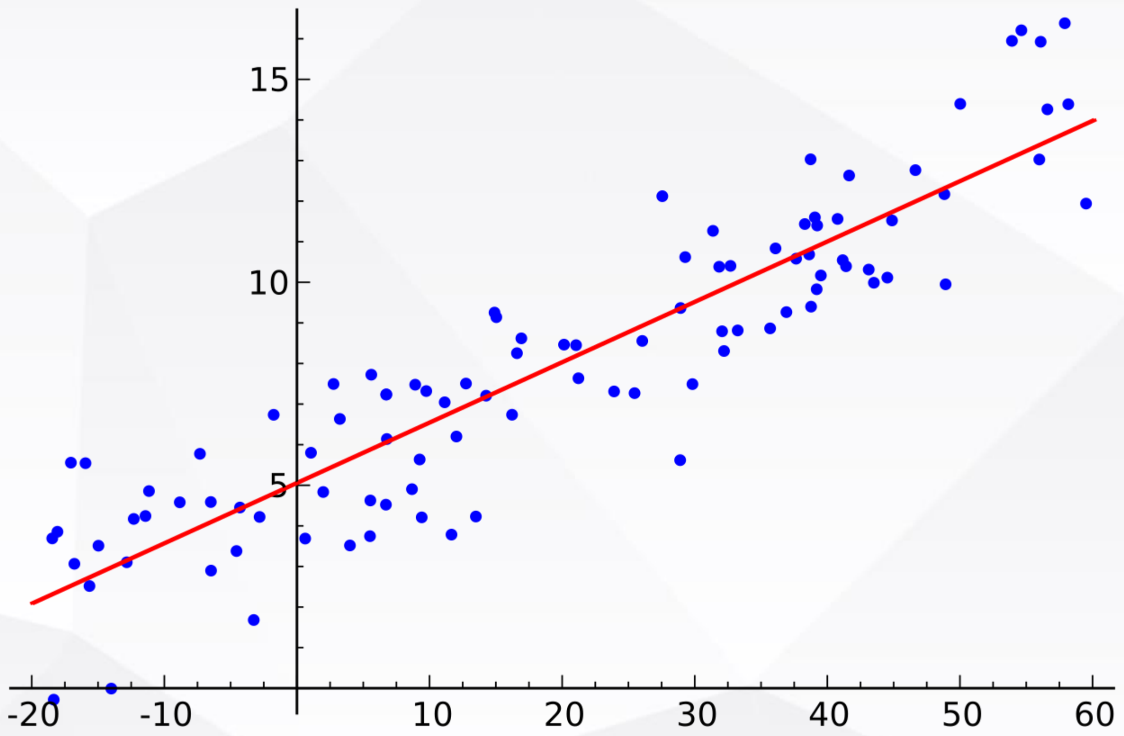
Regression analysis

$$Y_i = f(X_i, \beta) + e_i$$

We want a function *f* which takes our experimental settings (vector ***X***) and predicts our experimental outcome (*Y*) in the presence of experimental error *e.*

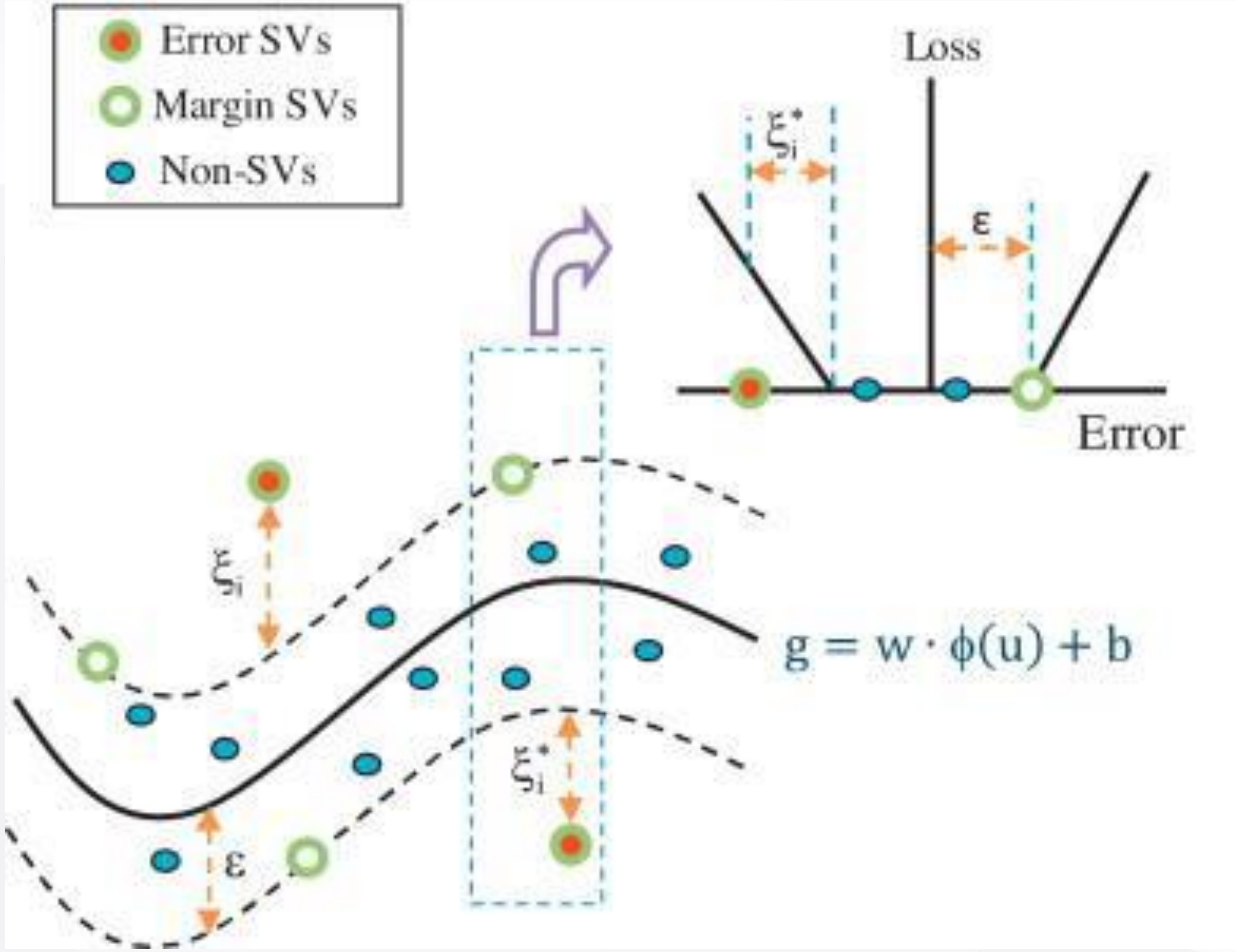
We also might be interested in β, which are parameters in the regression model.



Regression line for 50 random points in a Gaussian distribution around the line y=1.5x+2

en.wikipedia.org/wiki/Regression_analysis

Support vector machine regression



<u>Dimensions</u>

1 independent variable: line

2 independent variables: plane

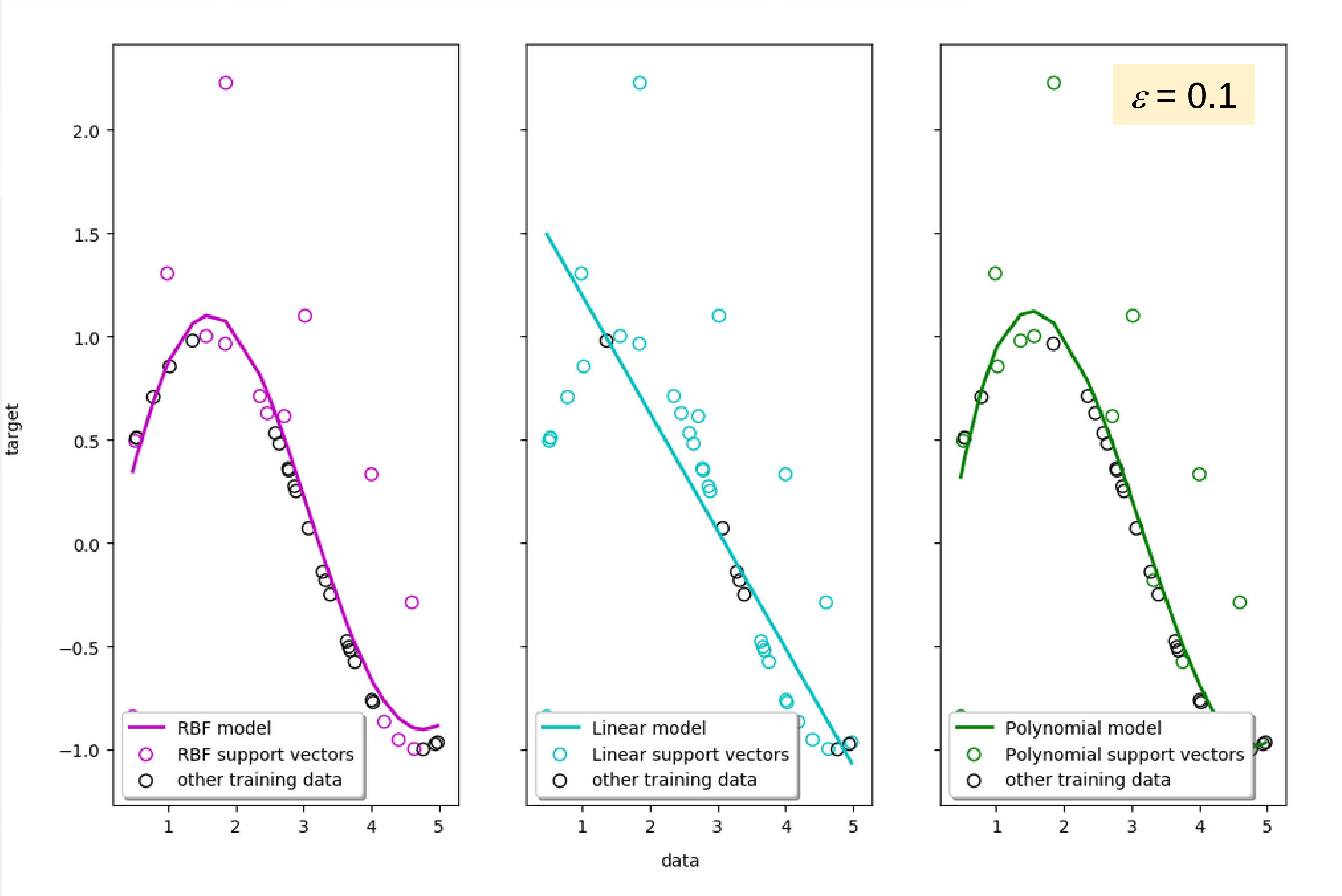
>2 independent variables: hyperplane

<u>Support vectors</u>

- Points within ε of the fit are "close enough"
- Only points $>\varepsilon$ away from the fit contribute the error (loss) function
- *Fitting consists of finding the hyperplane which puts as many points inside the ε tube as possible*

https://www.svm-tutorial.com/2014/11/svm-understanding-math-part-2/

SVR kernels



Linear	$k(\mathbf{x},\mathbf{x}') = \mathbf{x}^{\mathrm{T}}\mathbf{x}'$
Polynomial	$k(\mathbf{x},\mathbf{x}') = \langle \mathbf{x}\cdot\mathbf{x}' \rangle^{d}$
Gaussian	$k(\mathbf{x},\mathbf{x}') = \exp\left(-\frac{\lVert \mathbf{x}-\mathbf{x}' \rVert^{2}}{2\sigma^{2}}\right)$

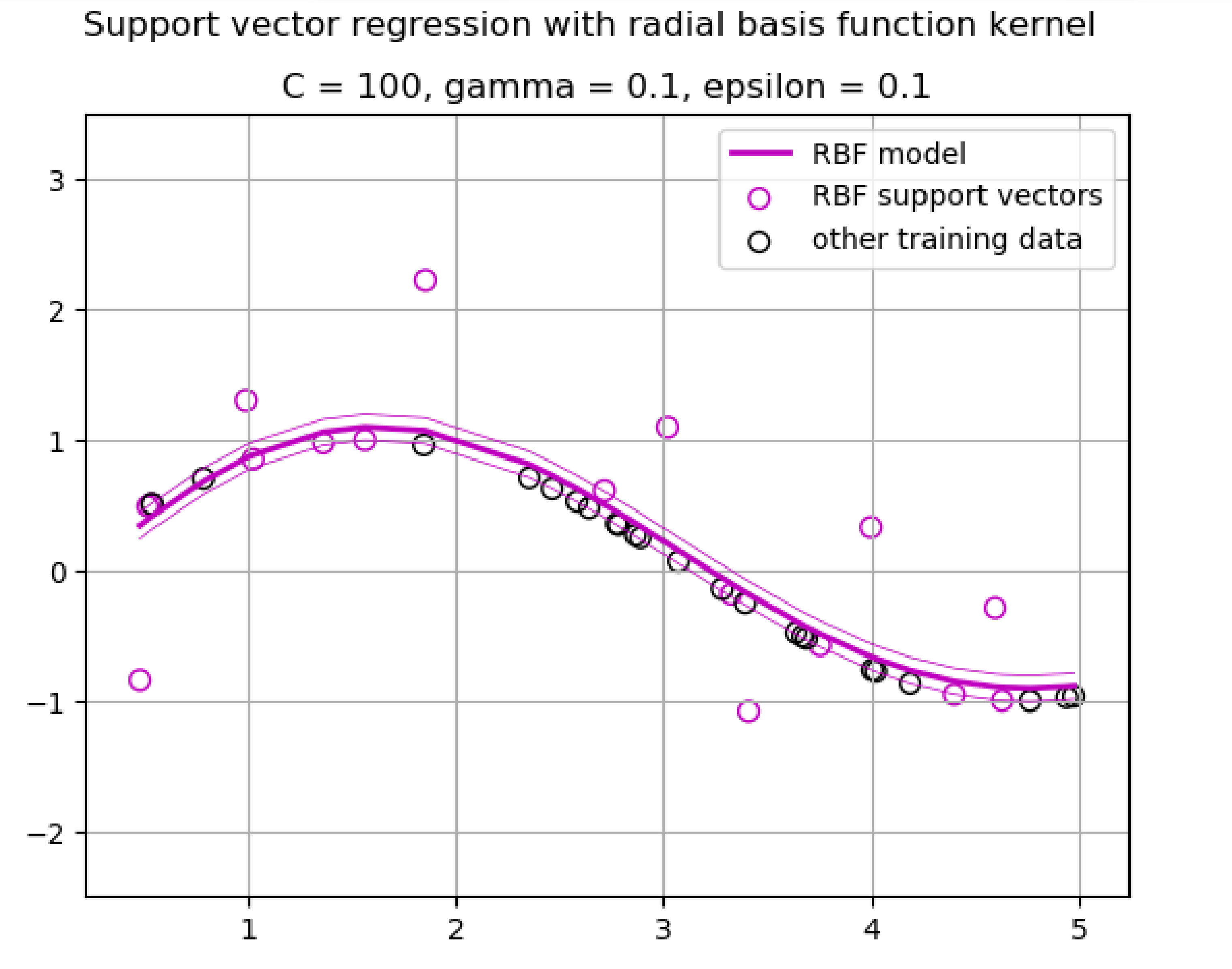
Kernels govern the shape of the fit function

Gaussian aka radial basis function is good for finding maxima and minima

https://www.svm-tutorial.com/2014/11/svm-understanding-math-part-2/

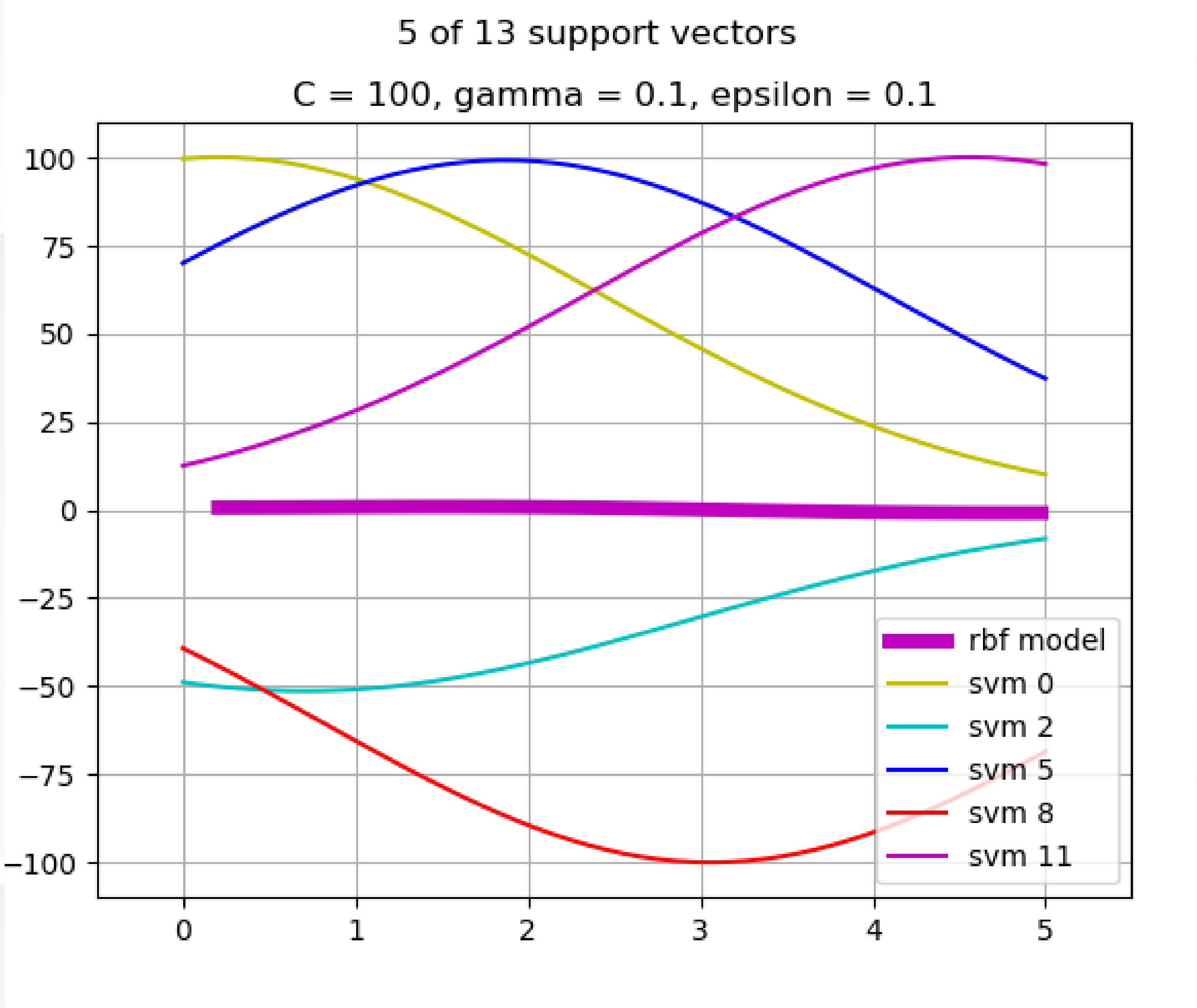
Support vectors and ε

$\exp(-\gamma(x - x')^2)$



SVR model: details

$$\exp(-\gamma(x - x')^2)$$



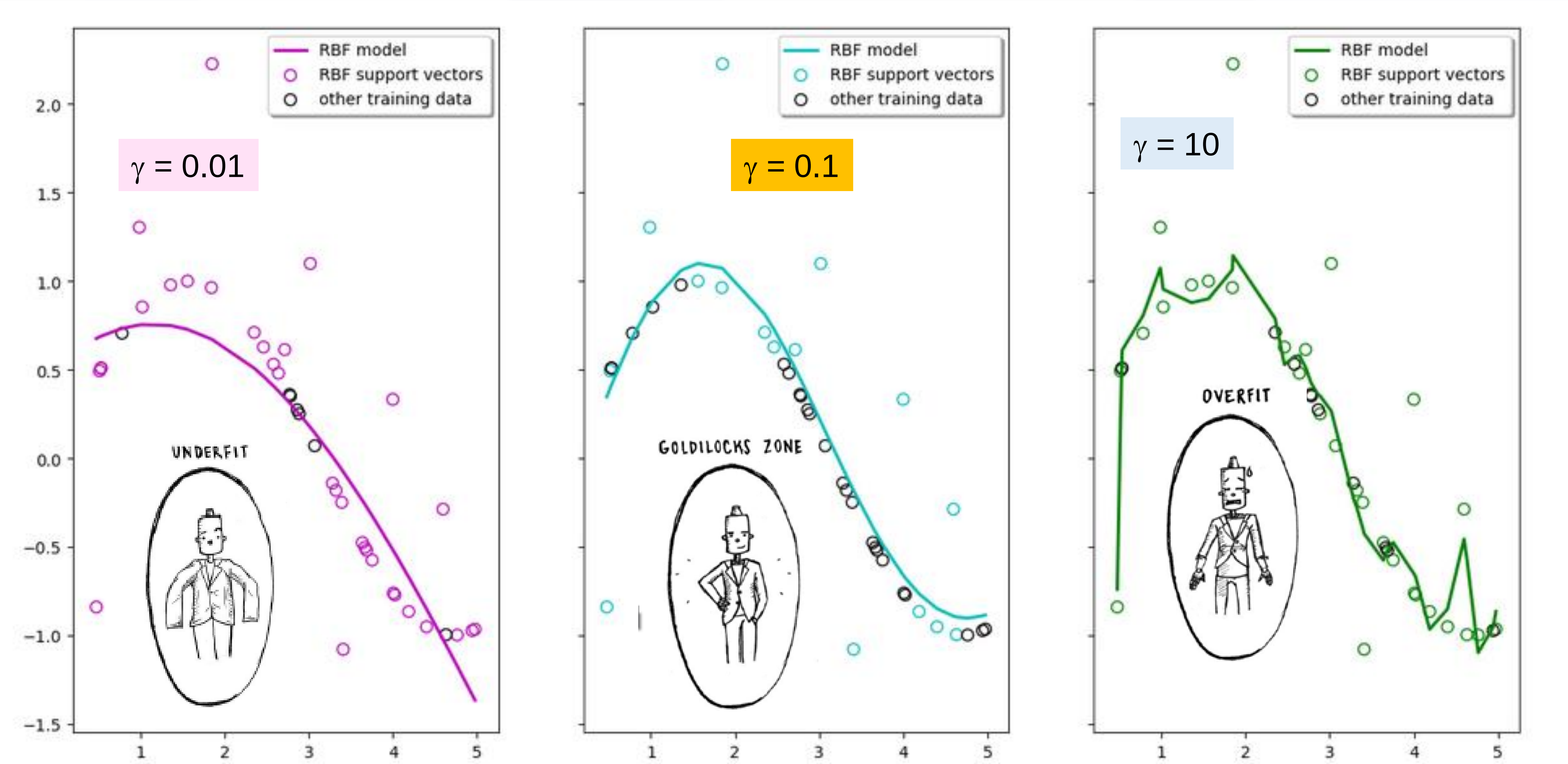
SVR fit is a sum of Gaussians centered on the support vectors with amplitudes between –*C* and +*C*

C = 100
ε = 0.1

Support vector machine

Overfitting and underfitting

$$\exp(-\gamma(x - x')^2)$$



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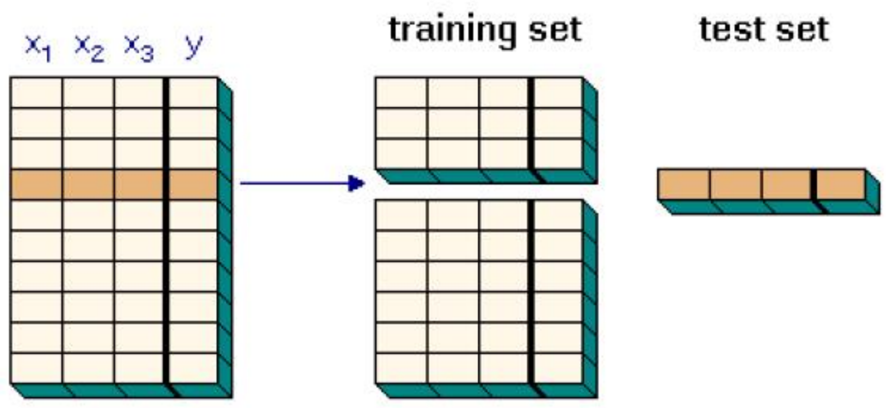
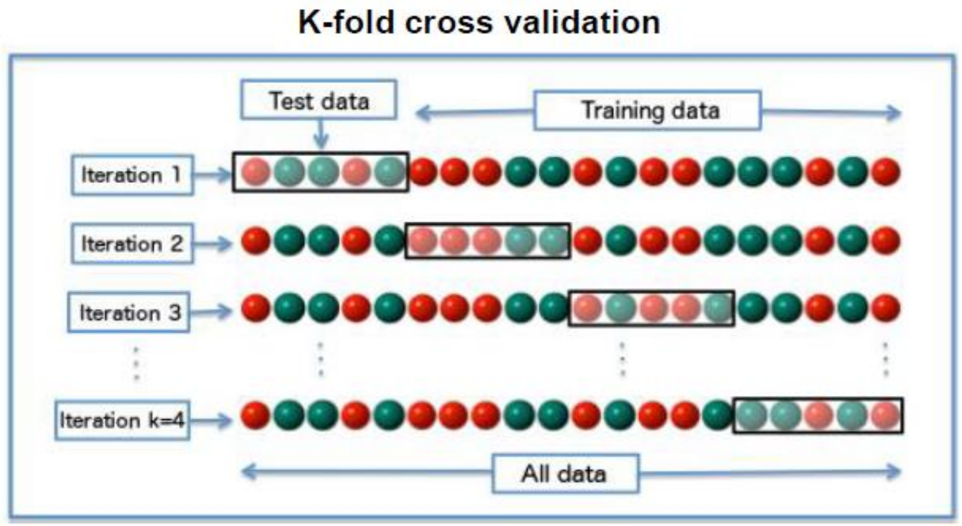
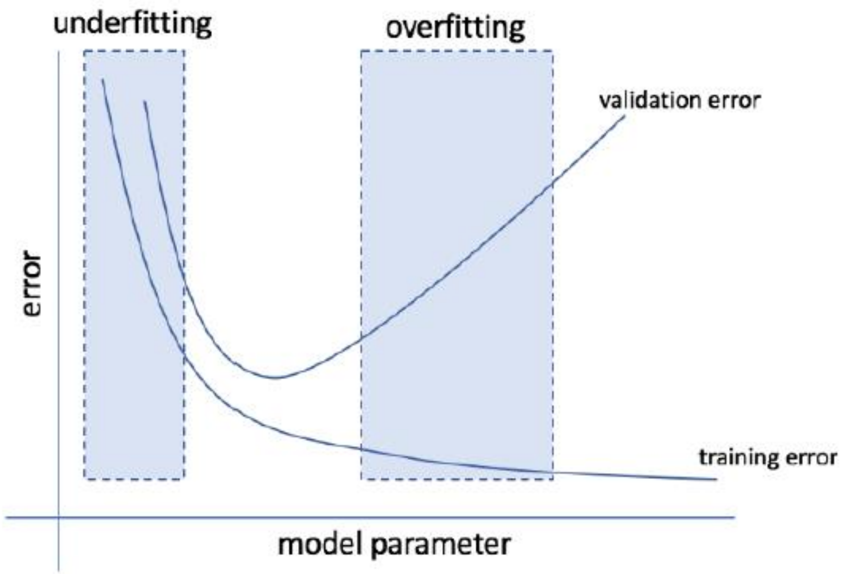
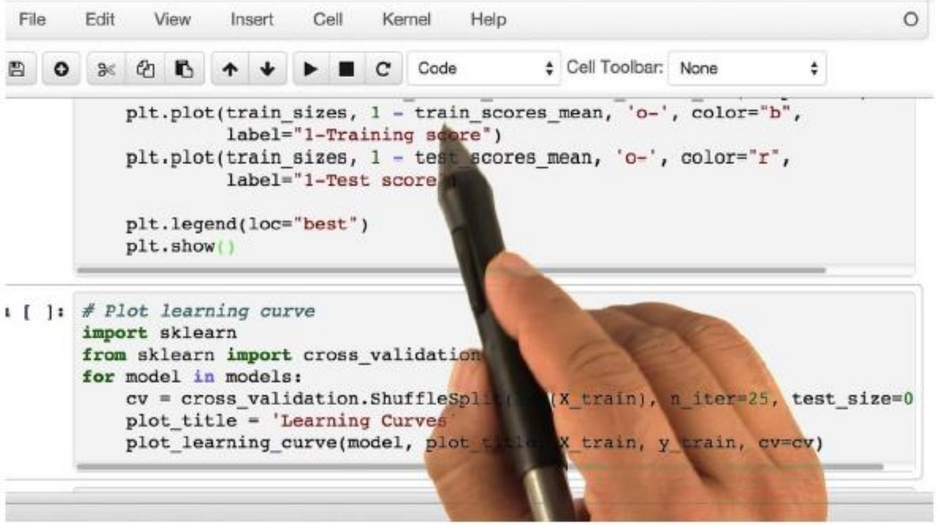
How to get the right fit?

Cross validation and learning curve





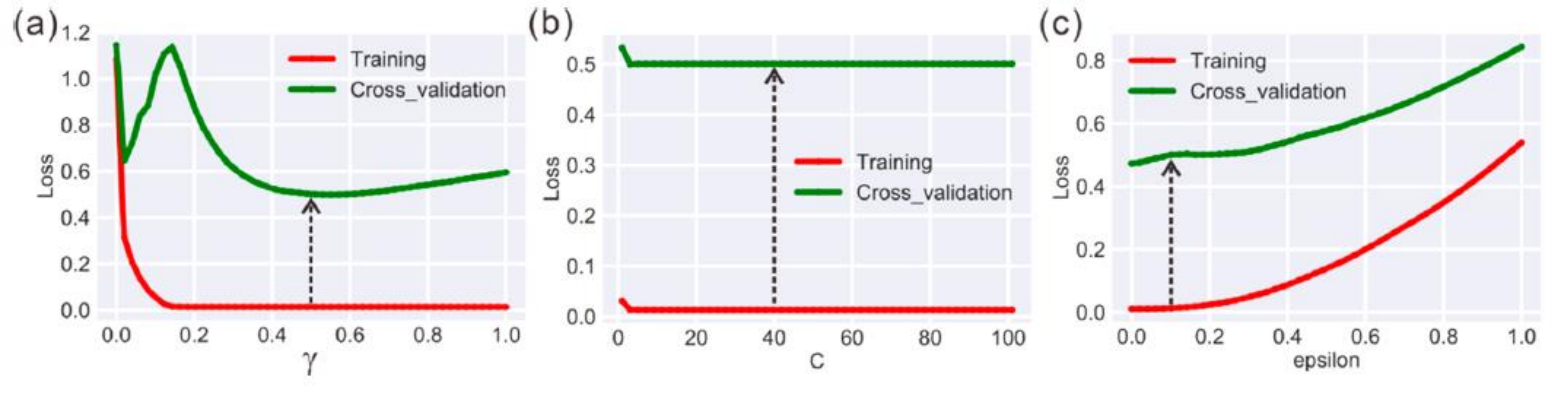




Machine Learning

Cross validation for first round of experiments

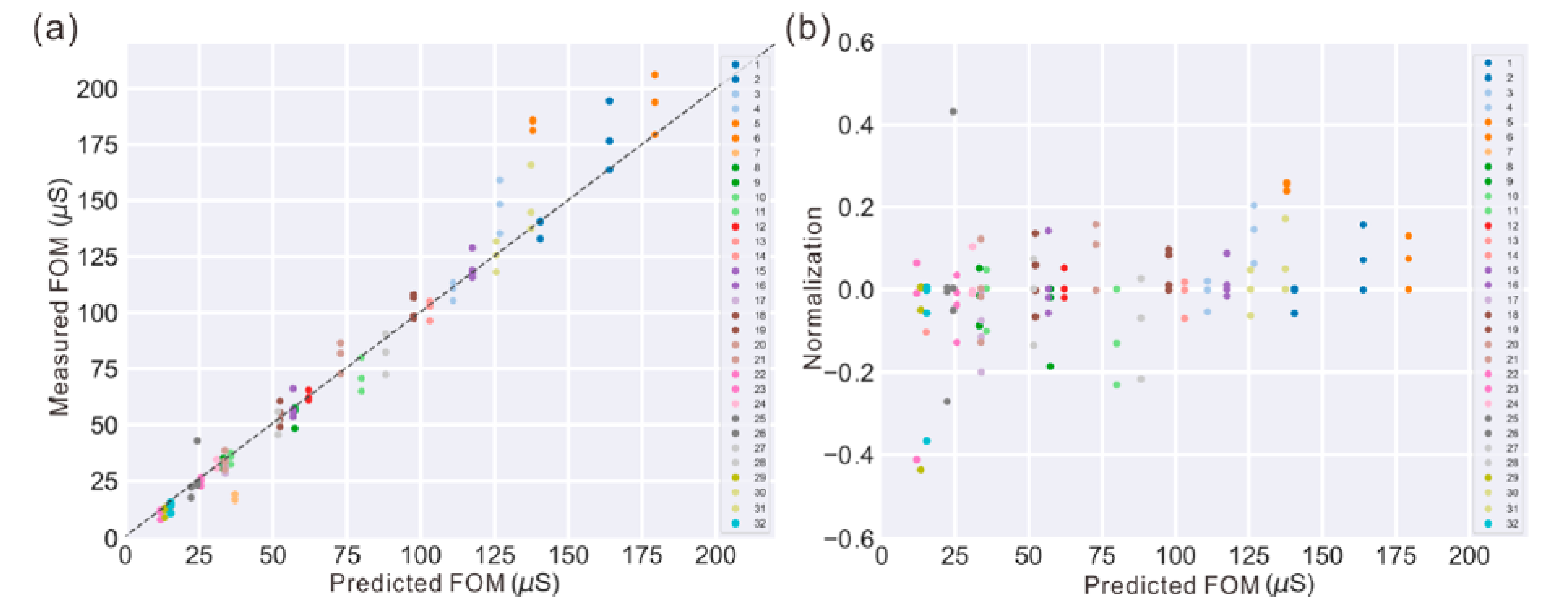
$$\exp(-\gamma(x - x')^2)$$



10 fold cross validation on the RBF hyperparameters γ, C, and ε.

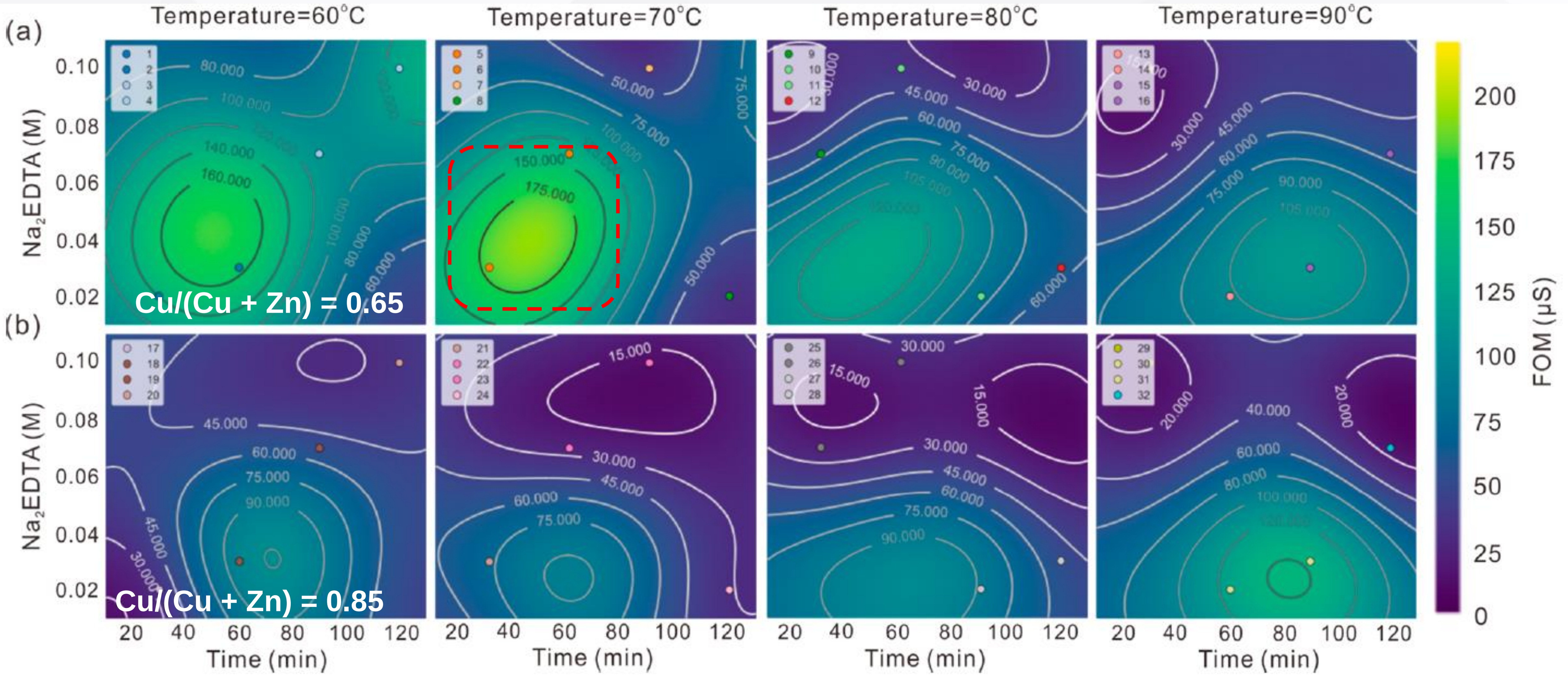
Wei, L.; Xu, X.; Gurudayal; Bullock, J.; Ager, J. W. Machine Learning Optimization of P-Type Transparent Conducting Films. *Chem. Mater.* **2019**, *31*, 7340–7350.

1st run SVR prediction



SVR applied to the first round: (a) measured vs. predicted FOM and (b) normalized results vs predicted FOM.

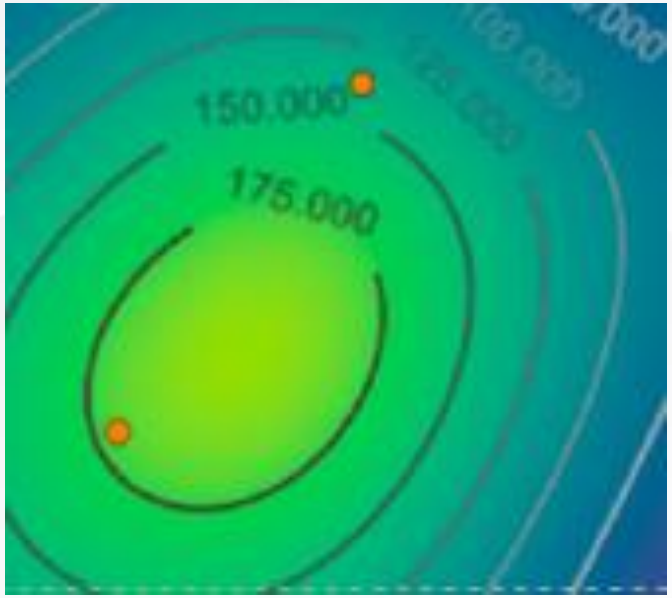
1st run contour maps



Wei, L.; Xu, X.; Gurudayal; Bullock, J.; Ager, J. W. Machine Learning Optimization of P-Type Transparent Conducting Films. *Chem. Mater.* **2019**, *31*, 7340–7350.

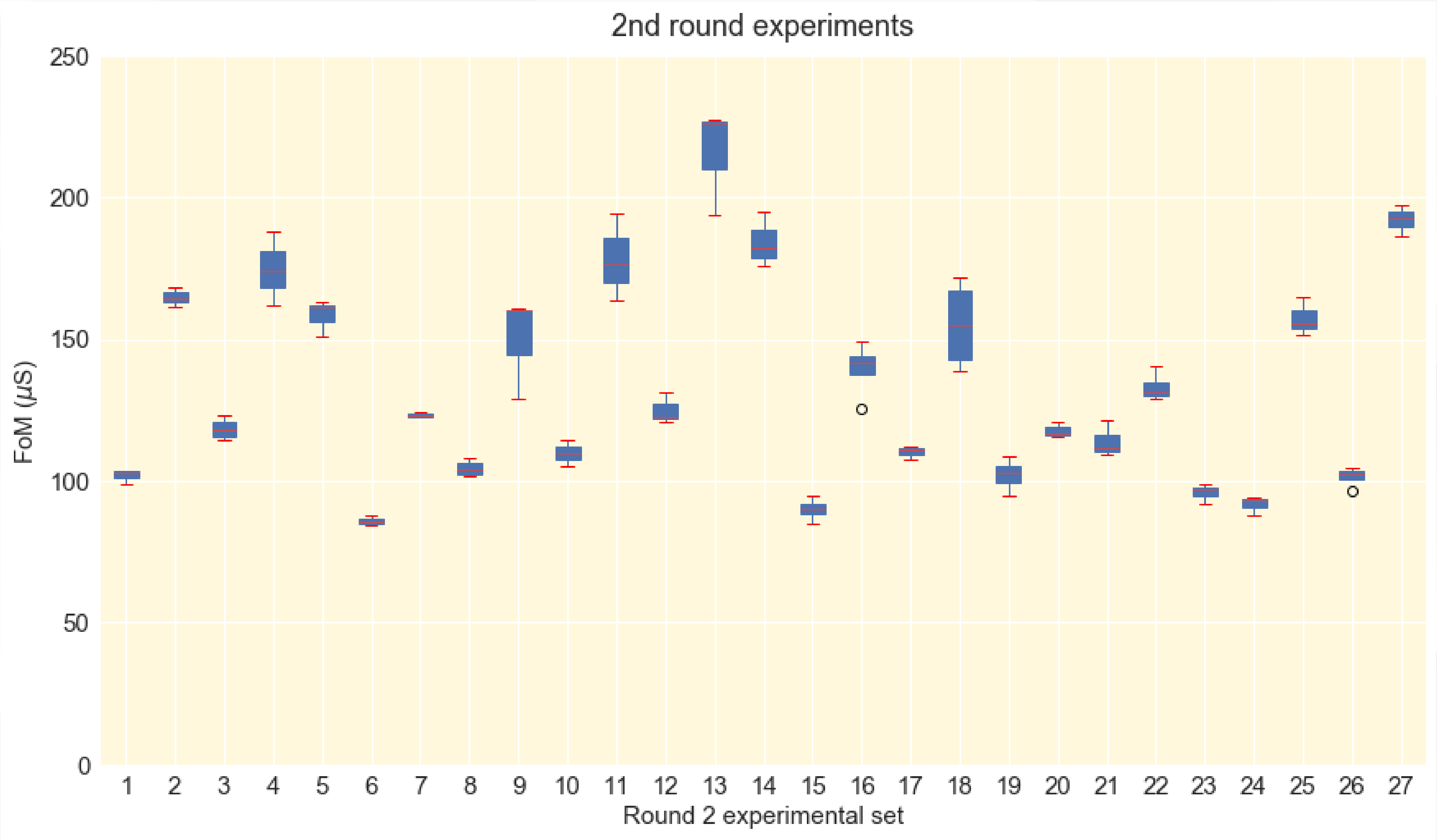
2^{nd} Run DoE

set no.	Cu/(Cu + Zn)	temperature (°C)	time (min)	concentration of Na_2EDTA (M)
1	0.6	60	50	0.05
2	0.6	60	60	0.03
3	0.6	60	70	0.04
4	0.6	70	50	0.03
5	0.6	70	60	0.04
6	0.6	70	70	0.05
7	0.6	80	50	0.04
8	0.6	80	60	0.05
9	0.6	80	70	0.03
10	0.65	60	50	0.05
11	0.65	60	60	0.03
12	0.65	60	70	0.04
13	0.65	70	50	0.03
14	0.65	70	60	0.04
15	0.65	70	70	0.05
16	0.65	80	50	0.04
17	0.65	80	60	0.05
18	0.65	80	70	0.03
19	0.7	60	50	0.05
20	0.7	60	60	0.03
21	0.7	60	70	0.04
22	0.7	70	50	0.03
23	0.7	70	60	0.04
24	0.7	70	70	0.05
25	0.7	80	50	0.04
26	0.7	80	60	0.05
27	0.7	80	70	0.03



- Cu content: 0.6, 0.65, 0.7
- Temperature: 60, 70, 80 ^{o}C
- Reaction time: 60, 70, 80 min
- Na_2EDTA concentration: 0.02, 0.03, 0.04 M

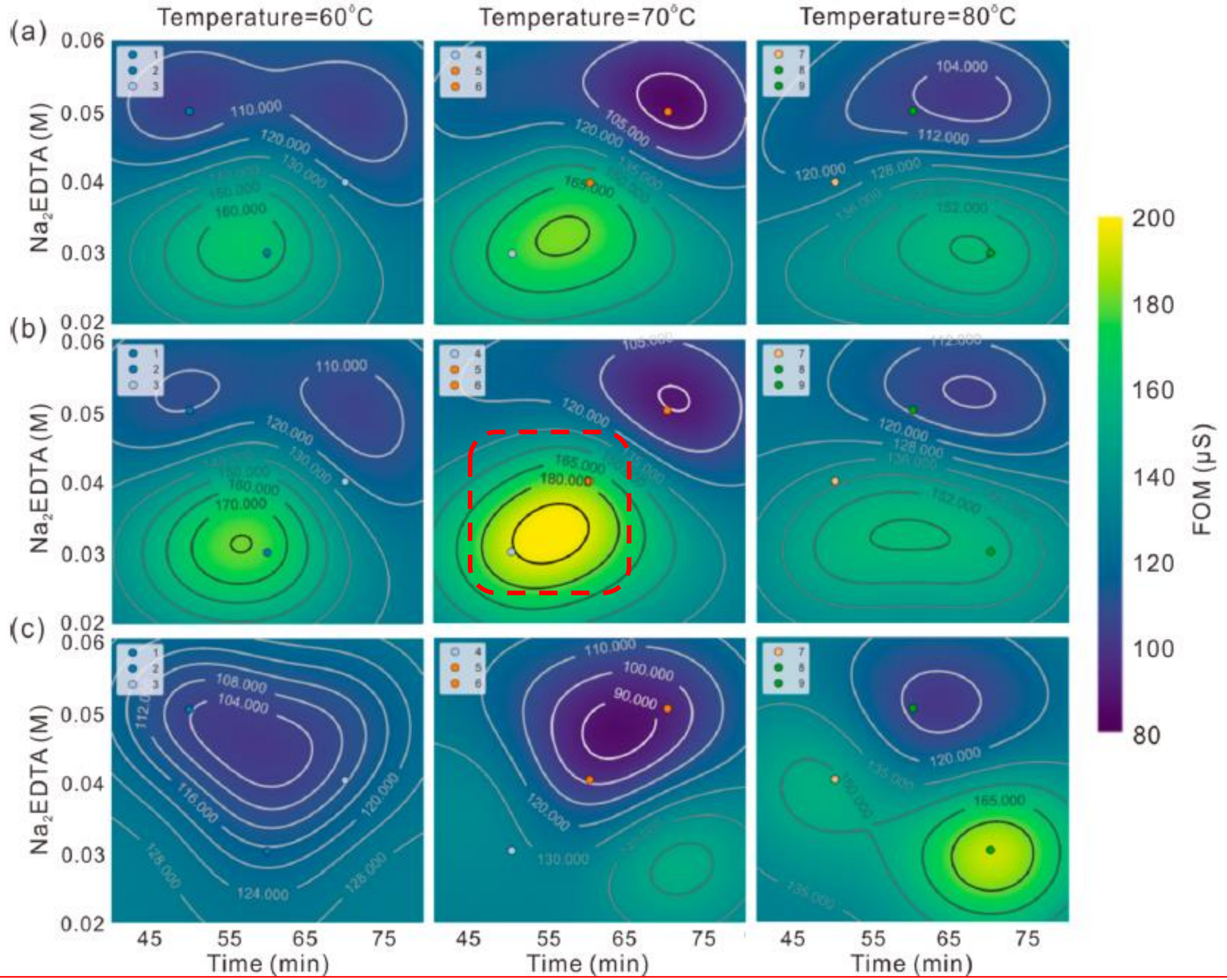
1/3 fractional factorial, balanced and orthogonal
27 Sets & 92 samples in total


2nd round experiments
FoM (μS)
250
200
150
100
50
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24
25
26
27
Round 2 experimental set

Cu/(Cu + Zn) = 0.60

Cu/(Cu + Zn) = 0.70

Cu/(Cu + Zn) = 0.80

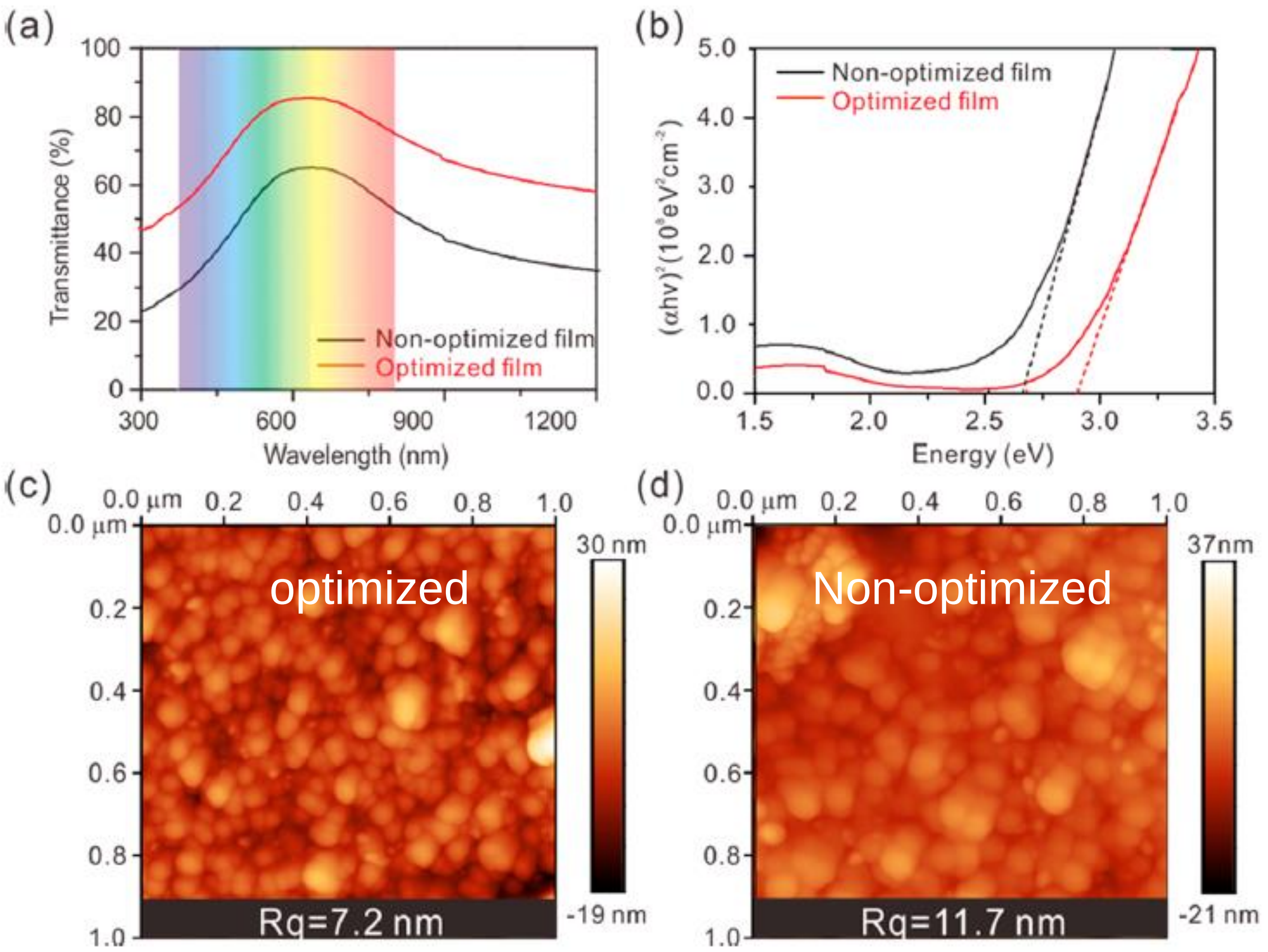


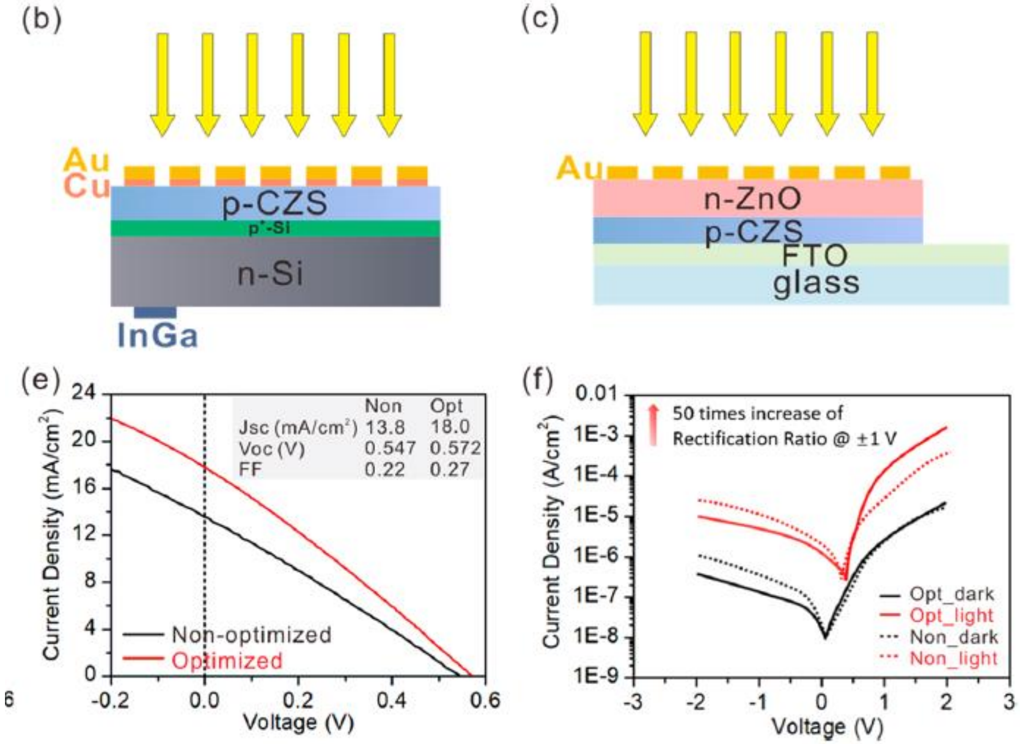
set no.	Cu/(Cu + Zn)	temperature (°C)	time (min)	concentration of Na_2EDTA (M)	FOM (μS)	no. of samples
13	0.65	70	50	0.03	216	3
14	0.65	70	60	0.04	184	3

Table 3. Comparison of Optical and Electrical Properties for Optimized and Nonoptimized Films[a]

film	Cu/(Cu + Zn)	temperature (°C)	time (min)	conc. of Na_2EDTA (M)	AVT (380−780 nm, %)	sheet resistance (Ω/□)	FOM (μS)
nonoptimized	0.85	80	60	0.1	0.57	529	14.8
optimized	0.65	70	60	0.03	0.75	363	173

[a]AVT, sheet resistance, and FOM are mean values of 10 samples with the same reaction condition.





Outline

Concepts

✓Design of Experiment (DOE)

✓Support Vector Machine Regression

✓Machine Learning and Cross-Validation

Resources

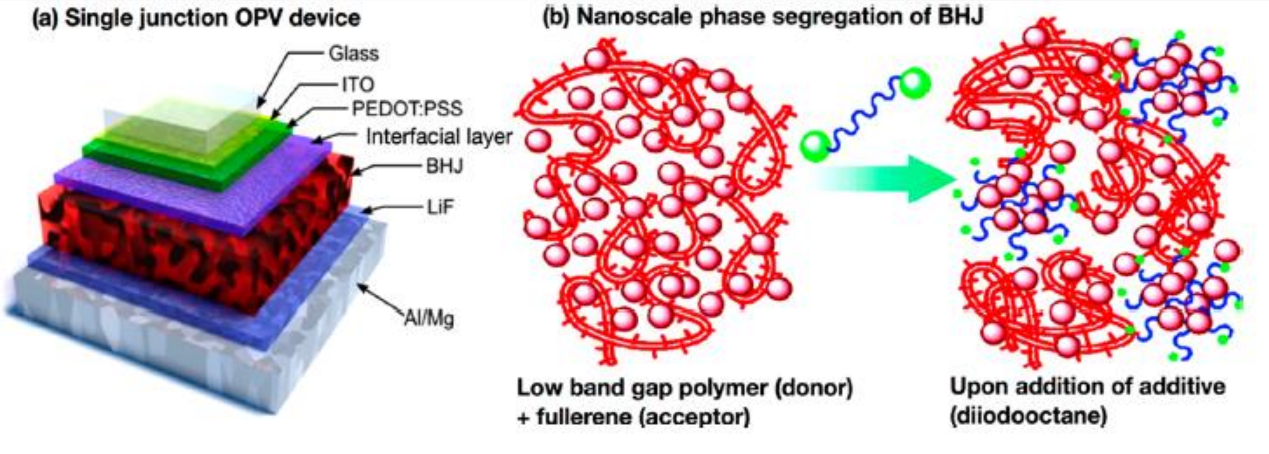
- Additional reading
- Open source software
- Commercial software

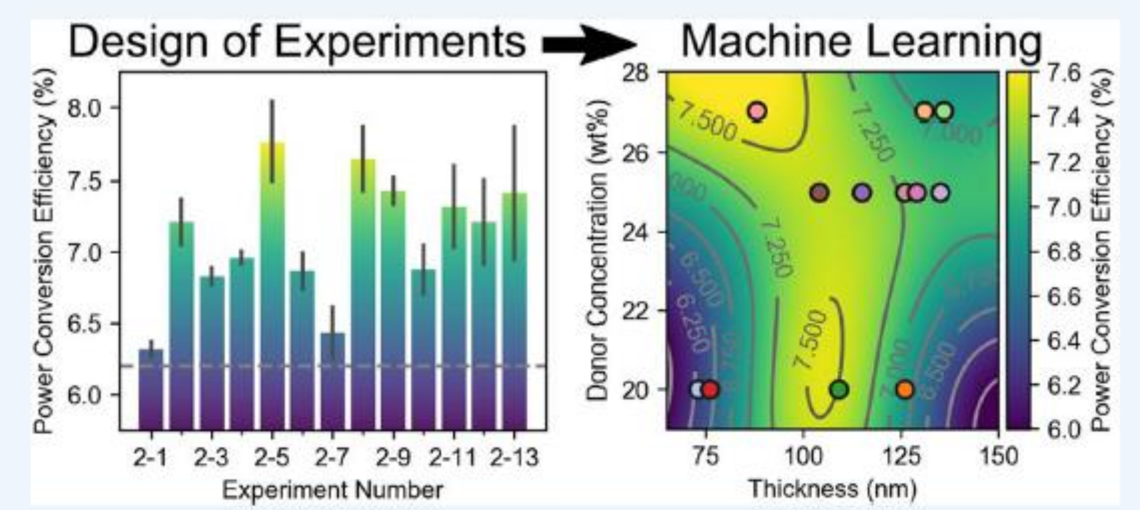
Case Studies

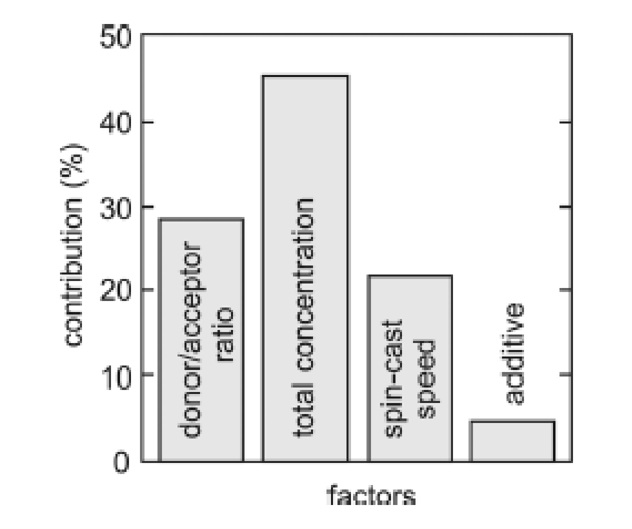
✓Simultaneous optimization of transparency and hole conductivity in solution-deposited CuZnS

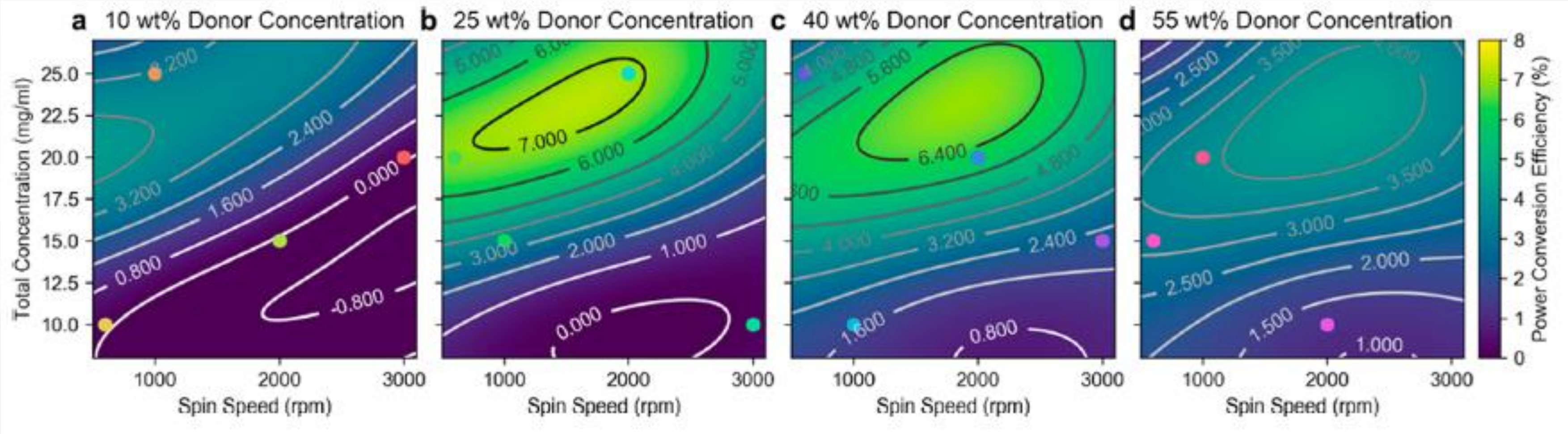
- Optimization of power conversion efficiency in solution-processed organic solar cells.

DOE and ML for OPVs









Cao, B.; Adutwum, L. A.; Oliynyk, A. O.; Luber, E. J.; Olsen, B. C.; Mar, A.; Buriak, J. M. How To Optimize Materials and Devices via Design of Experiments and Machine Learning: Demonstration Using Organic Photovoltaics. *ACS Nano* **2018**, *12*, 7434–7444.

Key Concepts

- Design of Experiment (DOE)
  Optimal experimental parameters (factors) in the least amount of time
- Analysis of Variance (ANOVA)
  Determine which factors affect outcome of experiment
- Support Vector Regression (SVR)
  Method to fit multi-dimensional data without the need for a physical model
- Cross-validation
  Machine learning method to find optimal hyperparameters

Outline

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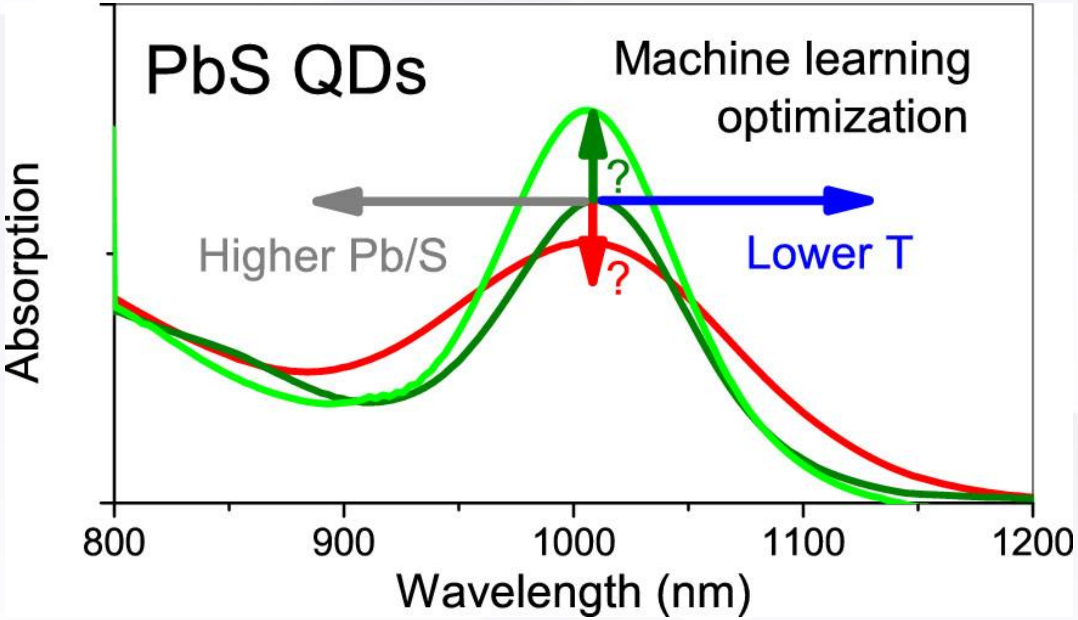
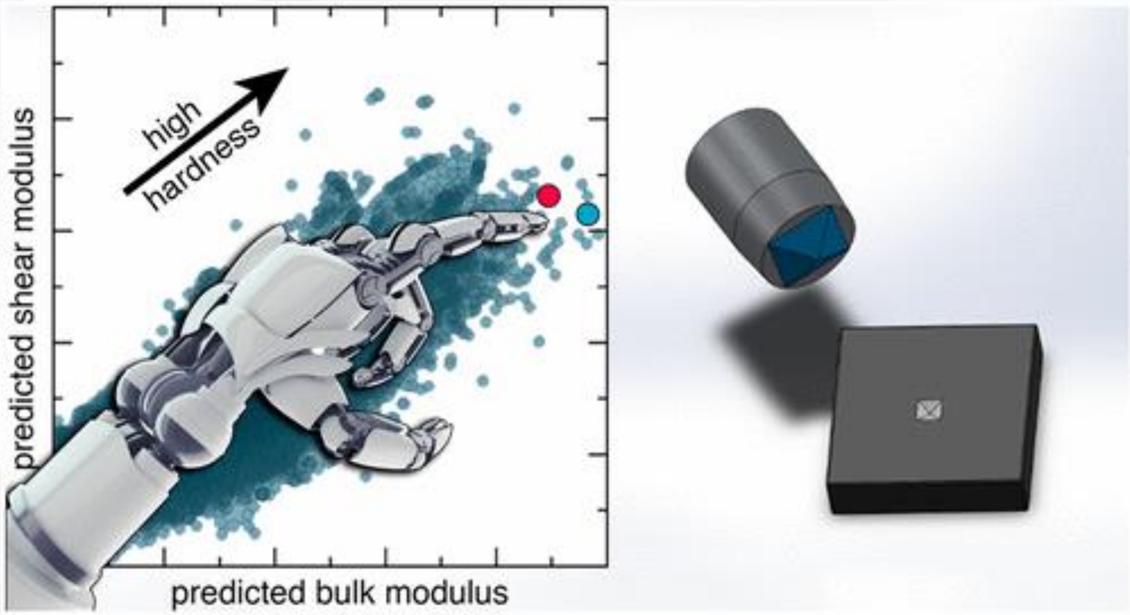
Additional Reading

Making mono-disperse quantum dots



Voznyy, O.; Levina, L.; Fan, J. Z.; Askerka, M.; Jain, A.; Choi, M.-J.; Ouellette, O.; Todorović, P.; Sagar, L. K.; Sargent, E. H. Machine Learning Accelerates Discovery of Optimal Colloidal Quantum Dot Synthesis. *ACS Nano* **2019**, acsnano.9b03864.

Discovering superhard materials



Mansouri Tehrani, A.; Oliynyk, A. O.; Parry, M.; Rizvi, Z.; Couper, S.; Lin, F.; Miyagi, L.; Sparks, T. D.; Brgoch, J. Machine Learning Directed Search for Ultraincompressible, Superhard Materials. *J. Am. Chem. Soc.* **2018**, *140*, 9844–9853.

nature materials

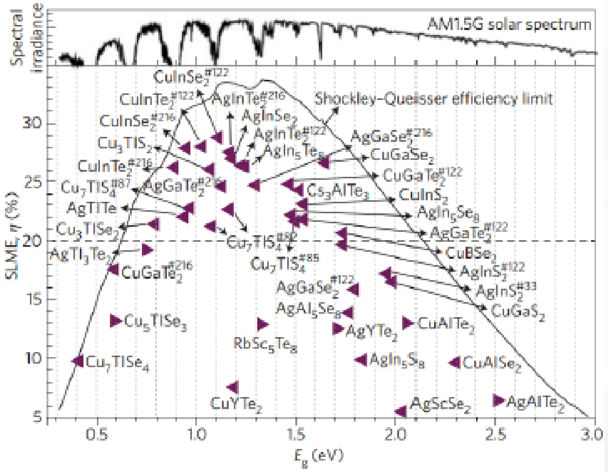
REVIEW ARTICLE

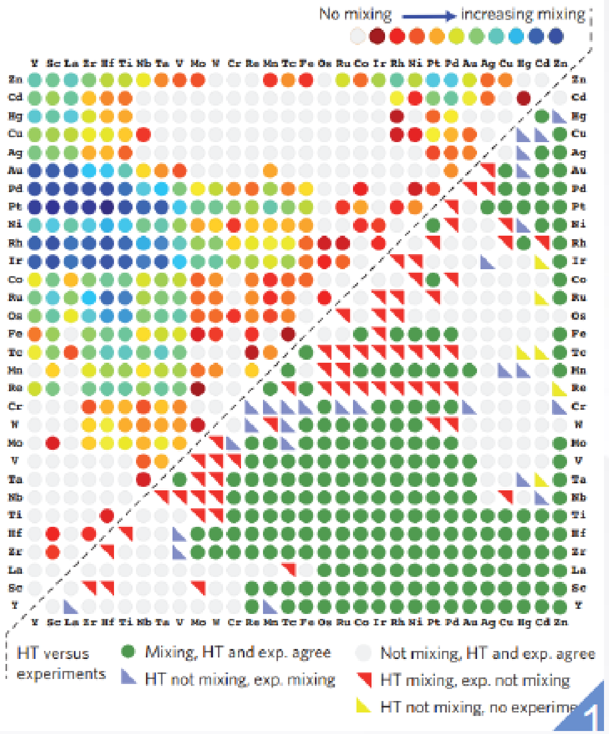
PUBLISHED ONLINE: 20 FEBRUARY 2013 | DOI: 10.1038/NMAT3568

The high-throughput highway to computational materials design

Stefano Curtarolo[1,2*], Gus L. W. Hart[2,3], Marco Buongiorno Nardelli[2,4,5], Natalio Mingo[2,6], Stefano Sanvito[2,7] and Ohad Levy[1,2,8]

Problem	Combination of materials properties (gene)	Descriptor
Structure stability: convex hull of an alloy system	Formation enthalpy (H_f) as a function of concentration (x) and the enthalpies (H) of A and B.	$H_f(x) = H(A_{1-x}B_x) - (1-x)H(A) - xH(B)$
Phase stability in off-lattice alloys	Spectral decomposition of alloy vector-energies ($E_{n,p}$, n-rows = species, p-columns = configurations) with principal-component-analysis coefficients (α_i) and truncation error ($\epsilon(d)$) (ref. 3).	$E_{n,p} = \alpha_1 E_{n,1} + \cdots + \alpha_{p-1} E_{n,p-1} + \epsilon(d)$
Nanosintered thermoelectrics	Ratio of the average power factor (<P>) to the grain size (L) (ref. 15).	$\hat{\chi}_{\mathrm{thermo}} \equiv \frac{<P>}{L}$
Topological insulators (epitaxial growth)	Variational ratio of spin-orbit distortion versus non-spin-orbit derivative strain (E_k^{SOC}, E_k^{noSOC}, spin/no spin-orbit bandgaps at k, a_0 lattice)[16].	$\hat{\chi}_{\mathrm{TI}} \equiv -\frac{E_k^{\mathrm{SOC}}(a_0)/a_0}{\delta E_k^{\mathrm{noSOC}}(a)_0/\delta a_0\big\|_{a_0}}$
Power conversion efficiency of a solar cell (spectroscopic limited maximum efficiency)	Ratio of the maximum output power density (P_m) to the incident solar energy density (P_{in}) — a function (η) of the radiative electron-hole recombination current (f_r) and the photon absorptivity ($\alpha(E)$) — versus bandgap energy (E_g)[62].	$\eta(\alpha(E), f_r) = P_m/P_{in}; E_g$
Non-proportionality in scintillators	Maximum mismatch between effective masses of electrons (m_e) and holes (m_h)[75].	$\hat{\chi}_{np} \equiv \max\left(\frac{m_e}{m_h}, \frac{m_h}{m_e}\right)$
Morphotropic phase boundary piezoelectrics	Energy proximity between tetragonal, rhombohedra and rotational distortions (ΔE_p). Angular coordinate (α_{AB}) of the energy minimum in the A-B off-centerings energy map for ABO_3 systems[79].	$\Delta E_p \leq 0.5$ eV $\alpha_{AB} \approx 45°$





Software resources – open source

Python

- Anaconda
- Machine learning packages:
  pandas, sklearn, tensorflow, keras
- Github:
  - JWA site
  - Lingfei Wei site
  - Design of Experiment, Tirthajyoti Sarkar

```
     Design-of-experiment builder by Dr. Tirthajyoti Sarkar, ON Semiconductor

                    June 2018, Sunnyvale, CA 94086
          Uses the following packages: numpy, pandas, pydoe, diversipy


------------------------------------------------------------
Design of Experiments menu
------------------------------------------------------------

1) Full factorial
2) 2-level fractional factorial
3) Plackett-Burman
4) Sukharev grid
5) Box-Behnken
6) Box-Wilson (Central-composite) with center-faced option
7) Box-Wilson (Central-composite) with center-inscribed option
8) Box-Wilson (Central-composite) with center-circumscribed option
```

R: DoE.base

(Full Factorials, Orthogonal Arrays and Base Utilities)

Grömping U (2018). "R Package DoE.base for Factorial Experiments." *Journal of Statistical Software*, **85**(5), 1–41.
doi: 10.18637/jss.v085.i05.

```
> oa.design(nlevels=c(2,2,2))
  A B C
1 1 2 2
2 1 1 1
3 2 1 2
4 2 2 1
class=design, type= oa
```

1/2 fractional factorial

Commercial Software

- JMP® 10 Design of Experiments Guide: https://www.jmp.com/support/downloads/pdf/jmp1001/doe_guide.pdf

Commercial Software

- JMP® 10 Design of Experiments Guide:

Popcorn DOE Resu...
Design Custom Design
Criterion D Optimal
Screening
Model
Constraint
DOE Dialog

Columns (5/0)
Brand
Time
Power
Number Popped
Total Kernels

	Brand	Time	Power	Number Popped	Total Kernels
1	Top Secret	3	7	30	420
2	Top Secret	3	7	120	420
3	Top Secret	3	10	120	400
4	Top Secret	4	8	250	430
5	Top Secret	5	5	370	420
6	Top Secret	5	5	400	420
7	Top Secret	5	8	350	420
8	Top Secret	5	8	370	380
9	Wilbur	3	7	374	400
10	Wilbur	3	8	340	440
11	Wilbur	3	10	20	410
12	Wilbur	4	6	440	460
13	Wilbur	4	9	340	400
14	Wilbur	5	5	170	370
15	Wilbur	5	7	370	380
16	Wilbur	5	8	420	450

scripts to analyze data

results from experiment

4 Factors
Space Filling Latin Hypercube

Factor Settings

Run	X1	X2	X3	X4
1	6.00000	6.00000	8.00000	5.00000
2	7.00000	7.00000	2.00000	6.00000
3	2.00000	5.00000	1.00000	4.00000
4	3.00000	2.00000	7.00000	2.00000
5	8.00000	3.00000	4.00000	3.00000
6	1.00000	4.00000	6.00000	8.00000
7	5.00000	1.00000	3.00000	7.00000
8	4.00000	8.00000	5.00000	1.00000

Design Diagnostics

Run	ScaledX1	ScaledX2	ScaledX3	ScaledX4	Minimum Distance	Nearest Point
1	0.71429	0.71429	1.00000	0.57143	0.821	5
2	0.85714	0.85714	0.14286	0.71429	0.782	5
3	0.14286	0.57143	0.00000	0.42857	0.833	2
4	0.28571	0.14286	0.85714	0.14286	0.845	1
5	1.00000	0.28571	0.42857	0.28571	0.782	2
6	0.00000	0.42857	0.71429	1.00000	0.845	7
7	0.57143	0.00000	0.28571	0.85714	0.782	5
8	0.42857	1.00000	0.57143	0.00000	0.821	1

discrepancy = 0.0397
Design Table
Make Table Back

Thank you