STRESS IN SU-8 PHOTORESIST FILMS: DOE APPROACH

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0305- 320 Design of Experiments

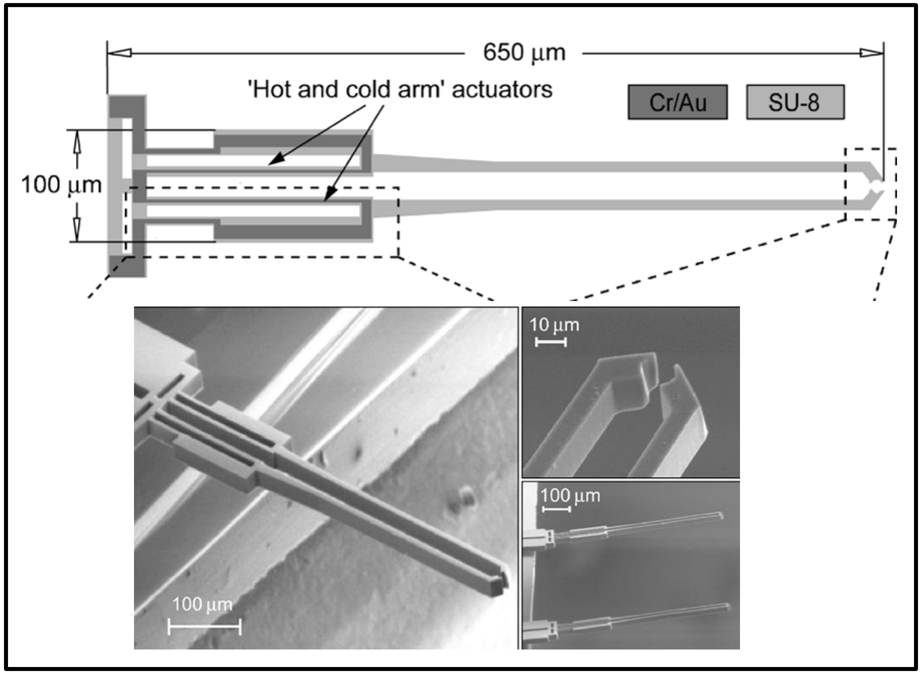
Overview

- Motivation
- Introduction
- Theory - Negative Photoresist
- Deposition Process
- Experimental Design
- Results and Analysis
- Conclusions
- Future Work

MOTIVATION

- SU-8 Photoresist is a common structural material for MEMS devices

Advantages: Biocompatibility, structural stability, chemically inert, lithographically patternable, low elastic modulus, hydrophobic



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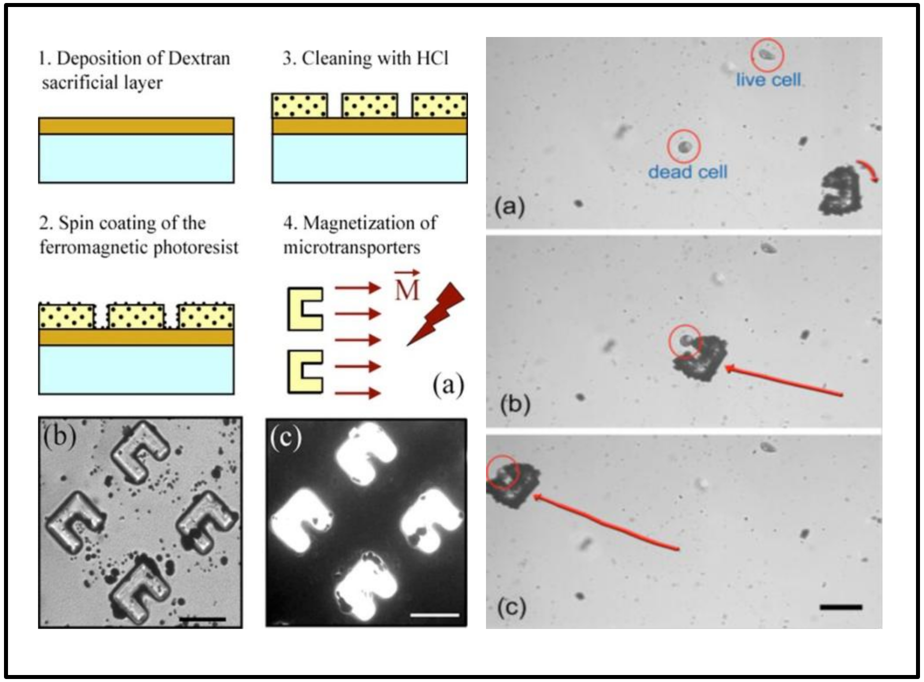

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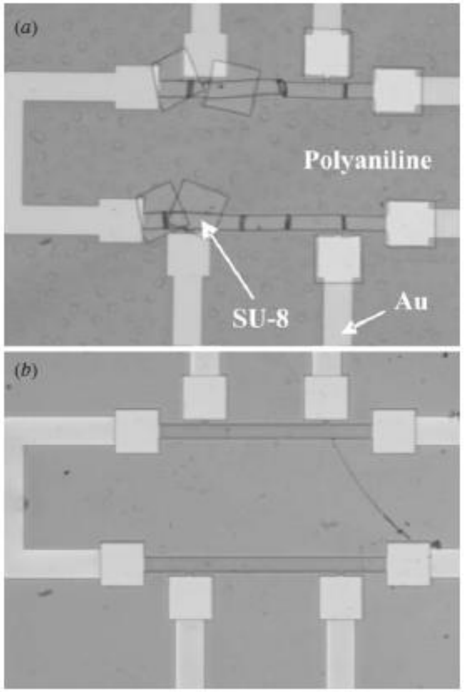
MOTIVATION



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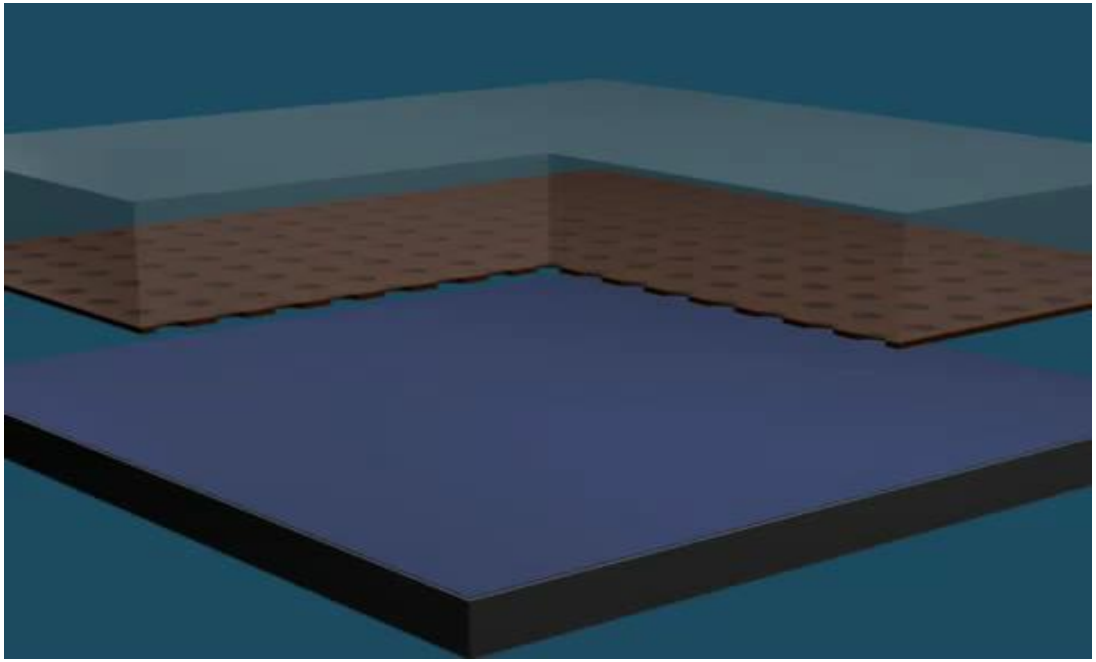
- Cracking or delamination due to the residual stress induced in the PR film material
- Might degrade the performance of the fabricated device significantly
- A through understanding and process optimization is necessary to tackle the problem

Theory- Negative Photo Resist

- Epoxy based, Negative tone PR
- *PAG compound* (triarylsulfoniumhexafluroantimonate), *EPON SU8 epoxy* (highly functionalized with 8 epoxy groups), *solvent* (γ-Butyrolacton)
- Cationic process is induced by PAG compound during UV illumination (PEB also accelerates the chemical reaction)



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Theory- Residual Stress

- Intrinsic and Extrinsic stress

Intrinsic stress: mostly generated during crosslinking due to the confinement of the monomers in the polymer matrix

Evaporation of solvent, loss of mass also result in intrinsic stress.

Extrinsic stress: involves the stress induced due to CTE mismatch (Si substrate and SU-8)

$$\sigma_{th} = (\alpha_{SU8} - \alpha_{si})\frac{E_{SU8}}{1 - \upsilon_{SU8}}(T_{PEB} - T_o)$$

α : CTE of the material

σ_{th}: Induced thermal stress

E_{SU8}: Young's modulus

T_{PEB}: PEB Temp

T_o: Ambient Temp

Theory- Residual Stress

- Stoney's Equation

$$\sigma = \frac{1}{6}\left(\frac{1}{R_{post}} - \frac{1}{R_{pre}}\right)\frac{E}{(1-\upsilon)}\left(\frac{(ts)^2}{(tf)^2}\right) \text{ , } Height = \frac{=[(\frac{Wafer\ dia}{2})^2]}{2R}$$

σ: stress in the film, after deposition

$Rpre$: substrate radius of curvature before deposition

$Rpost$: substrate radius of curvature after deposition

E: Young's modulus

υ: Poisson's ratio

ts: substrate thickness

tf: film thickness

E= 130 GPa
υ=0.279
ts= 650 μm

SU-8 resist Depositon Process

1) **Substrate Preparation**
   - Clean 6” [100] wafers.
   - No dehydration bake done.

2) **Manual Spin Coat**
   - Tool: SCS Resist Coater
   - Recipe : Two ramped levels rpm.

3) **Post Application Bake (PAB)**
   - Tool: Hot Plate
   - Recipe: Constant temperature.

4) **Exposure**
   - Tool: Karl Suss MA150 Contact Aligner
   - Recipe: Flood exposure with I-line.

5) **Post Exposure Bake (PEB)**
   - Tool: Hot Plate
   - Recipe: Constant temperature.

6) **Development**
   - Tool: Wet Chemistry
   - Recipe: PGMEA Puddle,
     IPA rinse,
     DI water rinse.
     Repeat if scrumming is visible.

7) **Hard Bake (HB)**
   - Tool: Hot Plate
   - Recipe: Constant temperature.

SCS Resist Coater

Karl Suss MA150

Wet Chemistry Bench

Hot Plates

Images Obtained From
http://wiki.smfl.rit.edu

Gathering Information

- Need to gain knowledge of the fabrication process.
- Used a set of suggested processing guidelines and ran a test process.
- Test process provides *knowledge* to *help* answer:
  - What factors are required?
  - Which factors are controllable?
  - What are the sources of noise?

Factors

Factors to Control

- RPM of spin coating
- PAB time
- Exposure dose
- PEB temp.
- PEB time
- Hard Bake (HB) temp.
- HB time

Factors to be Fixed

- Quantity of resist
- Spin time
- PAB temp.
- Quantity of developer
- Development time

Possible Noise Factors

- Ambient temp. and humidity
- Hot plate temp. variation
- Contamination
- Measurement noise

Goal and Objective

Goal

To minimize the residual stress in a film of SU-8 photoresist spin coated onto a bare silicon substrate.

Objective

To test the hypothesis that residual stress in a spin coated film of SU-8 photoresist onto a bare silicon substrate is a function of

- RPM
- PAB Time
- Exposure Dose
- PEB Temp
- PEB Time
- HB Temp
- HB Time

Fractional Factorial Design (2^{k-p})

- Number of Factors $k=7$
- Fraction: 1/8 $p=3$
- Number of Center Points 3
- Number of Treatment Combinations $n = 19$ (Full Factorial = 131)
- Generators
  - $E \approx ABC$ $F \approx BCD$ $G \approx ACD$
- Defining Contrast
  - $1 \approx ABCE, BCDF, ACDG, ADEF, BDEG, ABFG, CEFG$
- Confounding Pattern
  - $AB \approx CE, FG$
  - $AC \approx BE, DG$
  - $AD \approx EF, CG$
  - $AE \approx DF, BC$
  - $AF \approx DE, BG$
  - $AG \approx BF, CD$
  - $BD \approx CF, EG$

__Factor Mapping__

- A – HB Temp
- B – PEB Time
- C – Dose
- D – RPM
- E – HB Time
- F – PEB Time
- G – PAB Time

* If A and B are found to not interact: DG, DF, DE, and CD will be free of confounding

Factor Levels

TC Name	Factor	Low Level	High Level
A	HB Temp	175 °C	225 °C
B	PEB Temp	90 °C	95 °C
C	Dose	110 mJ/cm^2	140 mJ/cm^2
D	RPM	2500 rpm	3500 rpm
E	HB Time	10 minutes	20 minutes
F	PEB Time	3 minutes	4 minutes
G	PAB Time	2 minutes	3 minutes

- 1500 rpm was originally used
  - Thickness of the resist caused poor uniformity (expired material)
  - High spin coat rpm was needed.

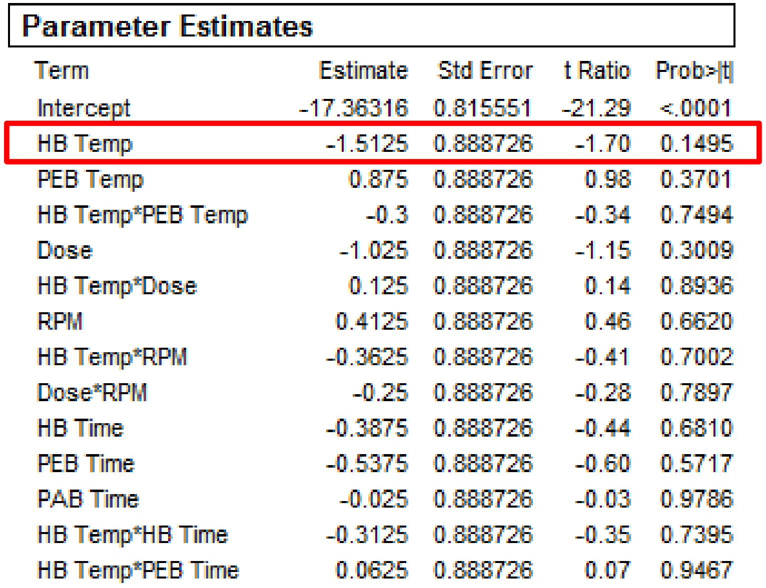
Results

Run Order	TC	HB Temp [°C]	PEB Temp [°C]	Dose [mJ/cm^2]	RPM	HB Time [minutes]	PEB Time [minutes]	PAB Time [minutes]	DEV Stress [MPa]	HB Stress [MPa]
19	0	200	95	125	2750	15	3.5	2.5	-7.14	-17.77
8	ab(fg)	225	100	110	2500	10	4	3	-5.08	-15.79
3	d(fg)	175	90	110	3500	10	4	3	-6.65	-15.21
9	b(ef)	175	100	110	2500	20	4	2	-6.30	-14.99
5	a(eg)	225	90	110	2500	20	3	3	-6.82	-19.94
1	bd(eg)	175	100	110	3500	20	3	3	-4.83	-11.31
13	bc(g)	175	100	140	2500	10	3	3	-7.52	-15.49
11	c(efg)	175	90	140	2500	20	4	3	-8.37	-18.73
7	cd(e)	175	90	140	3500	20	3	2	-6.61	-16.93
4	bcd(f)	175	100	140	3500	10	4	2	-7.78	-15.12
15	ad(ef)	225	90	110	3500	20	4	2	-6.41	-17.66
2	abc(e)	225	100	140	2500	20	3	2	-7.05	-17.50
6	-1	175	90	110	2500	10	3	2	-5.76	-15.46
16	0	200	95	125	2750	15	3.5	2.5	-12.97	-24.20
17	acd(g)	225	90	140	3500	10	3	3	-6.13	-17.65
10	ac(f)	225	90	140	2500	10	4	2	-7.75	-20.66
18	0	200	95	125	2750	15	3.5	2.5	-6.90	-17.26
19	abcd(efg)	225	100	140	3500	20	4	3	-9.41	-21.39
14	abd	225	100	110	3500	10	3	2	-6.40	-16.71

Analysis - Stress After Hard Bake

Main and 2-Factor

- Nothing Appears to be Significant.
- Possibly HB Temp

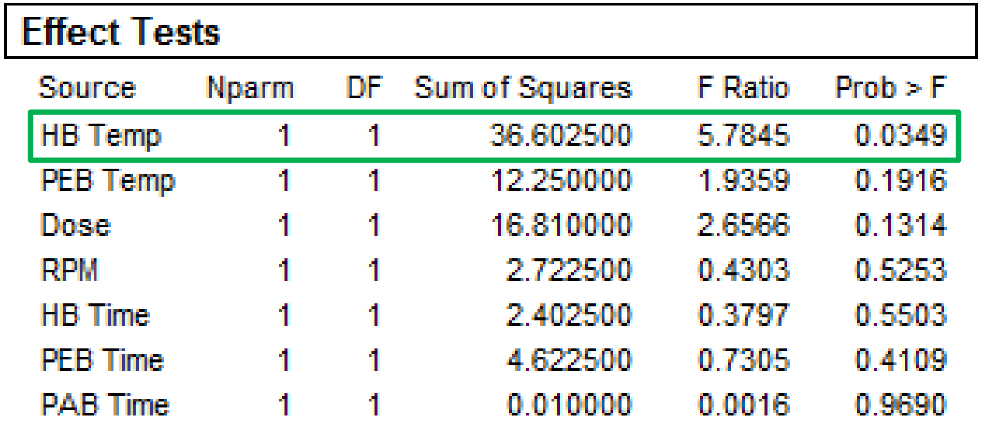


Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-17.36316	0.815551	-21.29	<.0001
HB Temp	-1.5125	0.888726	-1.70	0.1495
PEB Temp	0.875	0.888726	0.98	0.3701
HB Temp*PEB Temp	-0.3	0.888726	-0.34	0.7494
Dose	-1.025	0.888726	-1.15	0.3009
HB Temp*Dose	0.125	0.888726	0.14	0.8936
RPM	0.4125	0.888726	0.46	0.6620
HB Temp*RPM	-0.3625	0.888726	-0.41	0.7002
Dose*RPM	-0.25	0.888726	-0.28	0.7897
HB Time	-0.3875	0.888726	-0.44	0.6810
PEB Time	-0.5375	0.888726	-0.60	0.5717
PAB Time	-0.025	0.888726	-0.03	0.9786
HB Temp*HB Time	-0.3125	0.888726	-0.35	0.7395
HB Temp*PEB Time	0.0625	0.888726	0.07	0.9467

Main Factors Only

- HB Temp is the only significant effect.



Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
HB Temp	1	1	36.602500	5.7845	0.0349
PEB Temp	1	1	12.250000	1.9359	0.1916
Dose	1	1	16.810000	2.6566	0.1314
RPM	1	1	2.722500	0.4303	0.5253
HB Time	1	1	2.402500	0.3797	0.5503
PEB Time	1	1	4.622500	0.7305	0.4109
PAB Time	1	1	0.010000	0.0016	0.9690

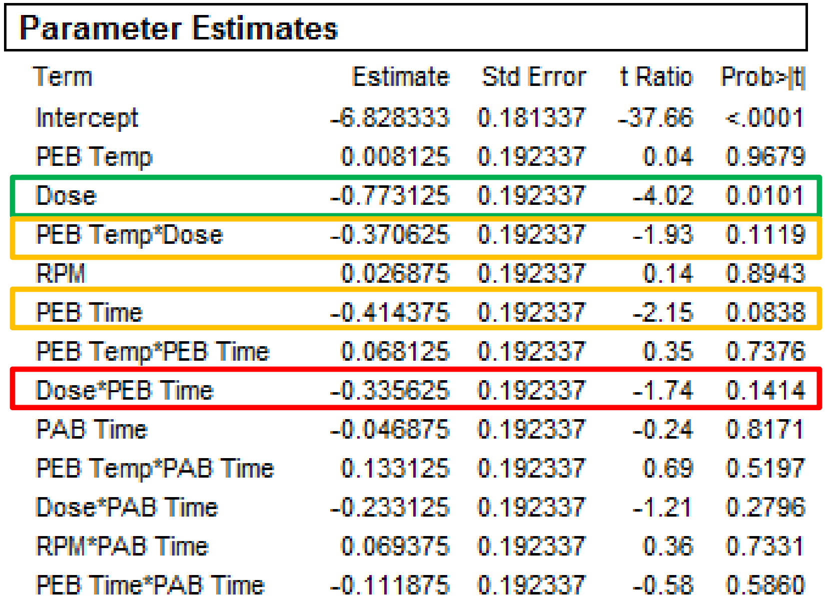
Analysis - Stress After Development

Main and 2-Factor

- α =0.05
  - Dose
- α =0.10
  - Dose, PEB Temp*Dose, PEB Time
- α =0.15
  - Dose, PEB Temp *Dose, PEB Time, Dose * PEB Time

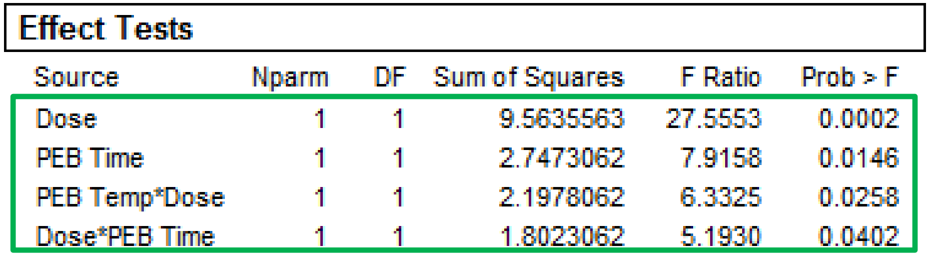
Significant Factors

- α =0.05
  - Dose
  - PEB Time
  - PEB Temp*Dose
  - Dose * PEB Time



Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-6.828333	0.181337	-37.66	<.0001
PEB Temp	0.008125	0.192337	0.04	0.9679
Dose	-0.773125	0.192337	-4.02	0.0101
PEB Temp*Dose	-0.370625	0.192337	-1.93	0.1119
RPM	0.026875	0.192337	0.14	0.8943
PEB Time	-0.414375	0.192337	-2.15	0.0838
PEB Temp*PEB Time	0.068125	0.192337	0.35	0.7376
Dose*PEB Time	-0.335625	0.192337	-1.74	0.1414
PAB Time	-0.046875	0.192337	-0.24	0.8171
PEB Temp*PAB Time	0.133125	0.192337	0.69	0.5197
Dose*PAB Time	-0.233125	0.192337	-1.21	0.2796
RPM*PAB Time	0.069375	0.192337	0.36	0.7331
PEB Time*PAB Time	-0.111875	0.192337	-0.58	0.5860

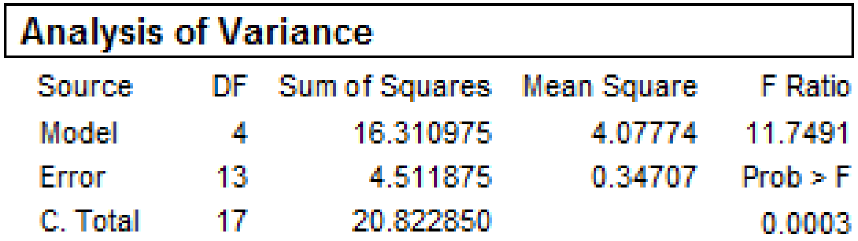


Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Dose	1	1	9.5635563	27.5553	0.0002
PEB Time	1	1	2.7473062	7.9158	0.0146
PEB Temp*Dose	1	1	2.1978062	6.3325	0.0258
Dose*PEB Time	1	1	1.8023062	5.1930	0.0402

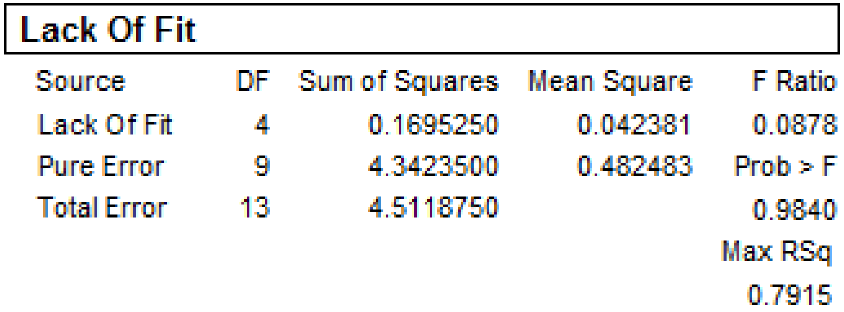
Analysis – Model (Development)

- Model is significant and of good fit



Analysis of Variance

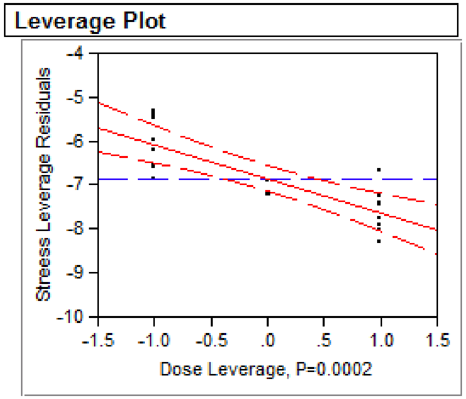
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	16.310975	4.07774	11.7491
Error	13	4.511875	0.34707	Prob > F
C. Total	17	20.822850		0.0003

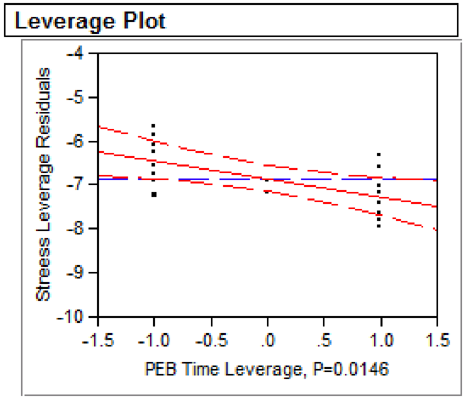


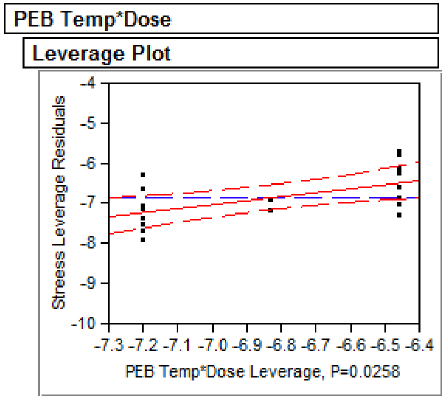
Lack Of Fit

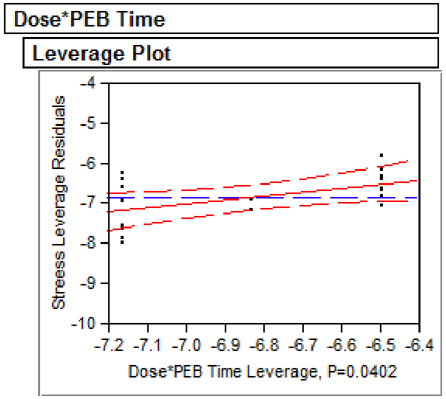
Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	4	0.1695250	0.042381	0.0878
Pure Error	9	4.3423500	0.482483	Prob > F
Total Error	13	4.5118750		0.9840
				Max RSq
				0.7915

- Leverage plot show effects and significance









Analysis - Confounding

- Confounding in significant effects
  - HB Temp * HB Time ≈ RPM * PEB Time , PEB Temp * Dose
  - PEB Temp * RPM ≈ Dose * PEB Time, HB Time * PAB Time
- No hard bake (Stress After Development)
- Based on prior knowledge, exposure and PEB should have an effect on stress due to shrinking caused by cross linking.
- Assuming above is true, confounded is resolved as:
  - PEB Temp * Dose
  - Dose * PEB Time

Analysis – Estimate of Response

- Estimate of Stress in design units

$$\hat{Y} = -6.83 - (0.77 * Dose) - (0.41 * PEB\_Time) - (0.37 * RPM * PEB\_Time) - (0.34 * HB\_Time * PAB\_Time)$$

- Optimum Factor Levels (Within high/low bounds)
  - *RPM = 2000
  - *PAB Time = 2 minutes
  - Dose = 110 mJ/cm^2
  - PEB Temp = 90 °C
  - PEB time = 3 minutes
  - *HB temp = 175 °C
  - *HB time = 10 minutes

$$\hat{Y} = -4.93 MPa$$

* Not used in the model equation, values set to minimum for conversation f time and energy.

Conclusion

- Unable to properly model stress after Hard Bake.
  - More knowledge is required on this processing step.
- Model was found for stress after development.
- Not all factors were found to be significant.
  - Dose, PEB Time, PEB Temp*Dose, Dose * PEB Time
  - Deconfounding of 2-factor effects is needed.
- From model and provided bounds
  - Minimum Stress -4.93 MPa
  - Larger bounds could yield lower stresses.
- Goal cannot be accessed without additional wafer to be processed.
- SU-8 is a very thick resist and challenging to work with.

Future Work

- Non-expired resist, wafers from the same batch
- Creating energy based factors i.e. Time*Temprature
- Processing the wafer with an optimum settings and measure the residual stress
- Running additional alpha start points to increase the levels and the range of the effects
- Fabrication of a test structure i.e. microcantilever, guckel rings in order to observe the residual stress effects

References

- [1] N. Chronis and L. Lee, “Electrothermally activated SU-8 Microgripper for Single Cell Manipulation in Solution,” JMEMS, vol. 14, pp. 857-867, 2005.
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- [4] http://sites.google.com/site/lergutierrez/su8_homepage