Residual Stress in SU-8 Photoresist Films

Ryan M. Bowen, Department of Microsystems Engineering, Rochester Institute of Technology

***Abstract*—SU-8 photoresist is a negative, epoxy-based, near-UV photoresist that is known for its ability to achieve large aspect ratios, good chemical resistance, and bio-compatibility. However, large internal stresses introduced during fabrication can cause cracking and delamination leading to potential device failure. Thus, what is presented is a designed screening experiment that concludes which process effects and interactions are significant with respect to residual stress. Using a designed fractional factorial experiment, significant factors are determined by performing an analysis of the means by using variances (ANOVA) with an alpha risk of 5% ($\alpha = 0.05$). Of all controllable process factors, a sub-set was considered including: spin coat RPM, Post Application Bake (PAB) time, exposure dose, Post Exposure Bake (PEB) temperature, PEB time, hard bake temperature, and hard bake time. Initial analysis of stress, measured after hard bake, concluded that the only significant factor was hard bake temperature. The unknown effects of the hard bake processing step resulted in analysis of stress only after development. The significant factors found to affect stress after development are exposure dose, PEB time, and the interactions of exposure dose with PEB temperature and PEB time. Based on an estimated model, derived by linear combinations of the significant effects, a low compressive stress of $4.93\,MPa$ can be achieved by using a low-level exposure dose ($110 mJ\backslash cm^2$), low-level PEB temperature ($90°$C) and and high-level PEB time (3 minutes). The results from this experiment lay ground for follow-up experiment(s) with the found significant effects.**

I. Introduction

THE negative epoxy-based SU-8 photoresist has been used in the fabrication of thick structures. High aspect ratios are achievable with SU-8 because of its low absorption coefficient at wavelengths above 300 nm. Patterning is possible for structures with millimeter range thickness and aspect ratios greater that 15. Other good features of SU-8 include high chemical resistance, biocompatability, and structural stability. However, SU-8 films are subject to cracking and delamination which in return can lead to device failure. Cracking and delamination are known to be associated with the residual stress in the SU-8 film introduced during processing. Therefore, if residual stress can be controlled and optimized, cracking and delamination can be avoided. Moreover, the residual stress in a SU-8 film on a silicon substrate is result of the combination of intrinsic and extrinsic stress introduced during processing. Intrinsic stress is driven by the cross-linking whereas extrinsic stress is related to thermal stress. From these two specific forms of stress, certain process factors will affect residual stress more as opposed to others. It is important to know what processing factors are significant and how they can be related to residual stress.

With an interest of using SU-8 photoresist to create Microelectromechanical Systems (MEMS) devices, structural stability is vital. Cracking and delamination onset by high residual stress can severely reduce structural stability. Therefore, the goal of the experiment is to optimize residual stress to reduce occurrences of cracking and delamination. Based on the goal, the objective of the experiment is to test the hypothesis that residual stress is a function of some determined process factors. The process factors are determined based on knowledge gained from executing a test treatment combination. Using only significant process factors, a model for residual stress can be created to estimate residual stress. With a precise estimate of residual stress it could be possible to find optimum process settings to target a specific residual stress that may be required for a device.

The layout of this paper follows with explanation of the theory behind residual stress in SU-8 films deposited on a silicon substrate. Additionally, how to measure/calculate residual stress in a film is discussed. Following the theory, is an outline of the experimental procedure including the process step in fabrication as well as the derivation of the treatment combinations for the designed experiment. From the designed experiment the stress response(s) and chosen process factors are analyzed to extract which effects are significant. The analysis of means using variances (ANOVA) technique is used to quantify effect significance with respect to a reasonable alpha risk. A model for residual stress is then constructed as a linear combination of the significant effects. The model is used estimate the residual stress given the process factors. Additionally, optimum factor settings can be extracted from the model. In concluded remarks, the results and analysis is summarized and future work explained.

II. Theory

A. Residual Stress

The residual stress in an SU-8 film after processing is composed of both intrinsic and extrinsic stress. The intrinsic stress is mostly generated by cross-linking during exposure. Cross-linking causes the film to be more dense causing shrinkage and essentially an intrinsic tensile stress. The extrinsic stress is related to externally forces or changes in ambient conditions. Thermal stress is the driving force for extrinsic stress, where stress is introduced because of mismatches in coefficient of thermal expansion (CTE). The thermal stress σ_{th} can be estimated as:

$$\sigma_{th} = (\alpha_{SU8} - \alpha_{Si}) \frac{E_{SU8}}{1 - \nu_{SU8}} (T_{PEB} - T_o) \quad (1)$$

where α_{SU8} and α_{Si} are the CTE of the two materials, E_{SU8} is the Young's modulus of SU-8, ν_{SU8} is Poisson's Ratio of the SU-8, T_{PEB} is the Post Exposure Bake (PEB) temperature and T_o is the ambient temperature.

Stoney's equation expresses the stress in a film deposited onto a substrate as a function of the material properties of the substrate, thickness of the substrate and film, and the radius of curvature of the substrate and film. Equation 2 is a representation of Stoney's Equation.

$$\sigma = \frac{1}{6}\left(\frac{1}{R_f} - \frac{1}{R_i}\right)\frac{E}{1-\nu}\frac{t_s^2}{t_f} \tag{2}$$

where E is the Young's modulus of the substrate, ν is Poisson's Ratio of the substrate, t_s is the substrate thickness, t_f is the film thickness, R_i is the initial radius of curvature of the substrate, and R_f is the radius of curvature of the substrate with deposited film. Profilometry can be used to measure the height of film/substrate as a function of position on the wafer. Assuming a circular, uniform, and symmetrical substrate/film the radius of curvature R can be approximated based on height as

$$R = \frac{r^2}{2\delta} \tag{3}$$

where r is the radius of the substrate, and δ is the height at the peak of the substrate curve.

B. Fractional Factorial Design

$$\#tc = 2^k \quad \text{Where } k = \#factors \tag{4}$$

1) Never confound single effects with each other.
2) Do not confound single effects with 2-factor interactions.
3) Do not confound 2-factor interactions with each other.

Fig. 1. Rules for confounding

III. Experimental Procedure

In this work, stress of a spin coated film of SU-8 photoresist on a silicon substrate is the response under analysis. To design a proper experiment, the required information needs to defined. Since the required information is determined based on some prior knowledge of the process, factors are chosen from those that are controllable during processing. Moreover, before designing the experiment knowledge of the process is required.

A. Fabrication Process

Listed below are the general processing steps, tools used, and controllable processing factors used to deposit a film of SU-8 photoresist on a bare silicon substrate.

1) Substrate Preparation: 6" [100] silicon wafers are cleaned, rinsed and dried and act as the substrate for the deposition of the SU-8 photoresist. No dehydration bake is performed to reduce the time of processing.

2) Spin Coat: SU-8 is manual deposited onto a bare silicon wafer and is then manually spin coated on a *SCS Resist Coater*. The spin coat recipe consists of two *RPM*s both having a *ramp-up time* and a *spin time*. The combination of the these spin speed ramps determine the thickness and uniformity of the SU-8 film.

3) Post Application Bake (PAB): After spin coating, the PAB (also referred to as a soft bake) is done using a hot plate at a chosen constant temperature and time. Thermal shock is avoided by slowly sliding wafer onto the hot plate.

4) Exposure: After PAB, the wafer is flood exposed using a *Karl Suss MA150 Aligner* with an I-line filter. The irradiance of the exposure tool is measured and exposure time varied to achieve a desired exposure dose.

5) Post Exposure Bake (PEB): After exposure, a PEB is done using a hot plate at a chosen constant temperature and time. Thermal shock is avoided by slowly sliding wafer onto the hot plate.

6) Development: The SU-8 photoresist is developed using a *puddling* technique where developer forms a meniscus on top of the wafer. The developer used is Propylene Glycol Methyl Ether Acetate (PGMEA) and development time controlled based on the thickness of the deposited SU-8 film. Immediately after development time expires, the wafer is rinsed with Isopropanal (IPA), rinsed with DI water, and dried with nitrogen. The IPA rinse process can leave scumming effects and when developing a pattern, repeated development and IPA rinse may be required.

7) Hard Bake: To further cross-link the SU-8 film a hard bake is done. The hard bake is achieved using a hot plate at a chosen constant temperature and time. Thermal shock is avoided by slowly sliding wafer onto the hot plate.

B. Design of Experiment

A test process is used to gain prior knowledge of the process as outlined above. From the test process, the controllable factors of the fabrication process are identified. Due to the large number of potential factors, some factors were chosen to be fixed. The fixed values were obtained from a recommended processing guideline. Table I summarizes the various sources of the process and their application to the experiment.

TABLE I
Chosen SU-8 film fabrication process factors.

Factor Name	Variable	Description
PRAmount		Photoresist deposition amount
RPM1		1^{st} RPM level
SpinTime1		1^{st} Spin time
SpinUp1		1^{st} Spin-Up time
RPM2	X	2^{nd} RPM level
SpinTime2		2^{nd} Spin time
SpinUp2		2^{nd} Spin-up time
PAB Temp		PAB temperature
PAB Time	X	PAB time
Dose	X	Exposure dose
PEB Temp	X	PEB temperature
PEB Time	X	PEB time
DevAmount		Amount developer used
DevTime		Development time
HB Temp	X	Hard bake temperature
HB Time	X	Hard bake time

As seen in Table I, there are seven factors chosen to be controlled in the experiment. If a full factorial experiment is considered, then 128 treatment combinations would be needed to run the experiment (Equation 4). Therefore, a more suitable number of treatment combination can be achieved using a fractional factorial design. Since this experiment is designed as a screening experiement, a large fraction of $\left(\frac{1}{4}\right)$ can be used, yielding a 2^{7-3} fractional factorial design requiring 16 treatment combinations. To improve the estimate of the residual error during analysis, three center points are collected, leading to a total of 19 treatment combinations required to run the experiment. To reduce violation of the rules of confounding (Figure 1) confounding was chosen according to Table II, where letters A-G represent the different factors in the experiment.

TABLE II
FRACTIONAL FACTORIAL DESIGN GENERATOR, DEFINING CONTRAST, AND CONFOUNDING PATTERN.

Generators		
$E \approx ABC$	$F \approx BCD$	$G \approx ACD$
Defining Contrast		
$1 \approx ABCE, BCDF, ACDG, ADEF, BDEG, ABFG, CEFG$		
Confounding Pattern		
$AB \approx CE, FG$ $AC \approx BE, DG$ $AD \approx EF, CG$	$AE \approx DF, BC$ $AF \approx DE, BG$	$AG \approx BF, CD$ $BD \approx CF, EG$

Based on the confounding pattern (Table II), if *A* and *B* are found not to interact then *DG*, *DF*, *DE*, and *CD* will be free of confounding. Therefore, *A* and *B* are chosen as factors least likely to interact resulting in factor order as in Table III.

TABLE III
FRACTION FACTORIAL DESIGN FACTOR MAPPING

Factor Letter	Physical Factor
A	HB Temp
B	PEB Time
C	Dose
D	RPM2
E	HB Time
F	PEB Time
G	PAB Time

IV. RESULTS AND ANALYSIS

All results are analyzed using the JMP IN software package. Once data is collected for the experiment, ANOVA tables are used to determine which effects are significant to estimate the response. Fitness of the model is then verified by analyzing the *lack of fit* of the model. The significant effects are assessed based on *p-values*, produced by JMP IN that correspond to F-Ratios, and a defined alpha risk.

After the test process is executed, the levels in which to run the experiment are selected as seen in Table IV. Before the levels were chosen, the test process was yielding poor uniformity during the spin coat process. This was determined to be caused by too low of a RPM and too long of ramp time in the second level of the spin coat recipe. Expired SU-8 photoresist was used and thus some of the solvent has evaporated causing the SU-8 to be more viscous requiring higher RPM and lower ramp times.

TABLE IV
FACTOR LEVELS FOR EXPERIMENT.

Letter	Factor	High Level	Low Level
A	HB Temp	175°C	225°C
B	PEB Temp	90°C	95°C
C	Dose	110 $mJ\backslash cm^2$	140 $mJ\backslash cm^2$
D	RPM2	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

TABLE V
MATERIAL PROPERTIES

Symbol	Property	Value
E	Young's Modulus substrate	130 GPa
ν	Possion's Ration substrate	0.279
t_s	Substrate thickness	77 mm
r	Substrate radius	650 μm

From the levels specified in Table IV, the treatment combinations (in physical and design units) for the fractional factorial design can be seen in the Appendix. The responses recorded are stress after development and after hard bake. The stresses are calculated using Equation 2 are done using the material properties in Table V and an assumption of film thickness. Film thickness was not specifically measured but is assumed to affect the estimate residual stress proportionally.

The initial goal of the experiment is reduce stress in a SU-8 film after all processing steps. However, once hard baked, only hard bake temperatures were found to be significant. Due to limited prior knowledge of the hard bake processing step, residual stress is measured only after development. The *p-values* for main and 2-factor effects are seen in Figure 2. From Figure 2, with all the 2-factor interactions included in the model significant effects are highlighted based on different alpha risk levels. It is important to not prematurely remove effects and thus an alpha risk of 0.15 is considered for significance testing. Therefore, main effects of *Dose*, *PEB Time* are significant and the interactions of *Dose***PEB Time* and *Dose***PEB Temp* are found to be significant. After removing all the not significant effects, *p-value* are calculated as shown in Figure 3. Additionally, the effects in Figure 3 are significant with an alpha risk of 0.05. Leverage plots of the reduced effects as shown in Figure 6 visually confirms that the effects are significant with all lines passing through the center.

The significance of the model is determined by analyzing the ANOVA table as provided in Figure 4. The *p-value* in Figure 4, states that the model is significant with a *p-value* less than 0.05.

The fitness of the model can be evaluated by observing the *lack of fit* of the model. For a model to be of good fit, then the *lack of fit* needs to be not significant. Figure 5, is the lack of fit for the model and it concludes that the model is of good

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

Fig. 2. Parameter estimates for model of stress given all main and 2-factor effects.

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

Fig. 3. Parameter estimates for model of stress with only significant effects.

fit with a high *p-value* for *lack of fit*.

Using only the significant effects, a linear model can be generated as a linear combination of the effects such as

$$\hat{Y} = C_0 + (C_1 * C) + (C_2 * F) + (C_3 * C * F) + (C_4 * C * B) \quad (5)$$

where C_i are the half-effect or parameter estimates for the linear model and C, F, and B are *Dose*, *PEB Time*, and *PEB Temp* respectfully. The coefficients found by JMP are listed in Table VI. Using the model equation (Equation 5) and half effects (Table VI) optimum setting for the factors can be chosen to target zero residual stress. Without going outside the high and low levels for each factor, Table VII summarizes the optimized factor levels where factors with (*) are not significant respect to residual stress and are thus set to minimize time and energy. Using Equation **??** with values from Table VII, an estimated residual stress of -4.93 MPa (compressive stress) is found. This value is consistent with other recorded residual stresses. From the model, it suggests that residual stress is a function of *Dose*, *PEB Time* and *PEB Temp*. Furthermore, to target zero residual stress low *Dose*, low *PEB Temp.* and high *PEB Time* is required. However, confounding does exist with the 2-factor interactions and additional experiment(s) are needed to fully de-confound the effects.

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

Fig. 4. ANOVA table for estimated model for stress.

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

Fig. 5. Lack of fit table for estimated model for stress

TABLE VI
HALF-EFFECTS OF LINEAR MODEL

Half-Effect	Value
C_0	-6.83
C_1	-0.77
C_2	-0.41
C_3	-0.37
C_4	-0.34

V. CONCLUSION

The goal of this experiment was to optimize residual stress in an SU-8 photoresist film process on bare silicon substrate. Additionally, the objective is to test the hypothesis that residual stress is a function of spin coat RPM, Post Application Bake (PAB) time, exposure dose, Post Exposure Bake (PEB) temperature, PEB time, hard bake temperature, and hard bake time. To achieve the objective, a fractional factorial design was used to define treatment combinations for data collection. The fractional factorial design reduced the number of required treatment combinations are the cost of confounding some of the effects. The final results concluded that residual stress

TABLE VII
OPTIMIZED FACTOR LEVELS FOR EXPERIMENT.

Letter	Factor	optimized Level
A	*HB Temp	175°C
B	PEB Temp	90°C
C	Dose	110 $mJ\backslash cm^2$
D	*RPM2	2500 RPM
E	*HB Time	10 minutes
F	PEB Time	3 minutes
G	*PAB Time	2 minutes

is a function of *Dose*, *PEB Time* and *PEB Temp*, where some de-confounding is needed to fully justify which factors contribute to the effect. The linear model estimate suggests that a 4.95 MPa of compressive stress can be achieved with low dose followed by a long low temp PEB. The overall goal, however is not yet validated where a final wafer is needed to compare to estimated response. Future work includes using a Central Composite Design (CCD) to investigate potential non-linear effects. Also factors are to represented based on energy functions such as time and temperature of a bake can be combined into one bake energy. Non-expired SU-8 should obtained to allow for slower RPM of spin coat and ensure proper material properties. Finally fabrication of some physical devices could be done with observations focused on cracking and delamination.

Appendix A
Treatment Combinations

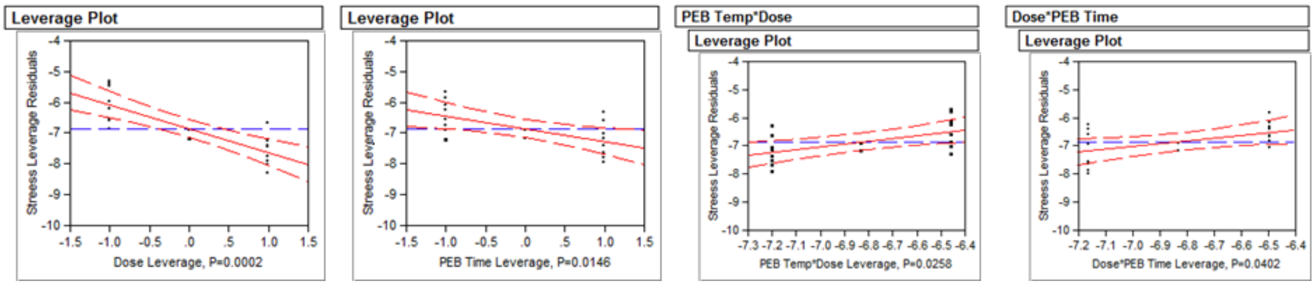

Fig. 6. Leverage plots for significant effects in experiment.

Run Order	TC	HB Temp [°C]	PEB Temp [°C]	Dose [mJ/cm²]	RPM	HB Time [minutes]	PEB Time [minutes]	PAB Time [minutes]	DEV Stress [MPa]	HB Stress [MPa]
19	0	0	0	0	0	0	0	0	-7.14	-17.77
8	ab(fg)	+	+	-	-	-	+	+	-5.08	-15.79
3	d(fg)	-	-	-	+	-	+	+	-6.65	-15.21
9	b(ef)	-	+	-	-	+	+	-	-6.30	-14.99
5	a(eg)	+	-	-	-	+	-	+	-6.82	-19.94
1	bd(eg)	-	+	-	+	+	-	+	-4.83	-11.31
13	bc(g)	-	+	+	-	-	-	+	-7.52	-15.49
11	c(efg)	-	-	+	-	+	+	+	-8.37	-18.73
7	cd(e)	-	-	+	+	+	-	-	-6.61	-16.93
4	bcd(f)	-	+	+	+	-	+	-	-7.78	-15.12
15	ad(ef)	+	-	-	+	+	+	-	-6.41	-17.66
2	abc(e)	+	+	+	-	+	-	-	-7.05	-17.50
6	-1	-	-	-	-	-	-	-	-5.76	-15.46
16	0	0	0	0	0	0	0	0	-12.97	-24.20
17	acd(g)	+	-	+	+	-	-	+	-6.13	-17.65
10	ac(f)	+	-	+	-	-	+	-	-7.75	-20.66
18	0	0	0	0	0	0	0	0	-6.90	-17.26
19	abcd(efg)	+	+	+	+	+	+	+	-9.41	-21.39
14	abd	+	+	-	+	-	-	-	-6.40	-16.71

Fig. 7. Treatment combinations and stress responses in design units

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

Fig. 8. Treatment combinations and stress responses in physical units