Investigation of Residual Stress on SU-8 Films: A Design of Experiments Approach

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ABSTRACT

This paper presents the results of an experiment designed to investigate significant factors affecting residual stress induced in thin SU-8 films. SU-8 material is a negative tone epoxy-based photoresist that is widely used in fabrication of Micro-Electro-Mechanical Systems (MEMS) devices usually as a structural material. Seven main factors are selected for a screening experiment utilizing the fractional factorial design approach. The fractionalization element is three and additional three treatment combinations are included for center points in order to obtain a better estimate of residual error. Residual stress is measured both before and after the hard bake step. With a 5 % alpha risk; dose, post exposure base time and their interactions are found to be significant factors. The data is analyzed using JMP software and related statistical results are also included.

Keywords: Residual stress, SU-8 resist, Design of Experiments

1. INTRODUCTION

The negative tone epoxy based SU-8 photoresist has been a common material preferred in fabrication of MEMS devices due to various advantages. It is a biocompatible material that might advantageous for bio-MEMS applications [1]. It is chemically inert and structurally very stable after cross-linking step [2]. Although it might be highly advantageous to use this material in various microsystems applications, a thorough understanding of processing knowledge is needed in order to obtain desired results.

One of the biggest problems in processing thin-films that are used as structural materials for MEMS devices is controlling the residual stress levels induced in the films. An uncontrolled stress level might result in a failure of a working device in the micro scale or might highly degrade its performance. Figure 1 below shows the effect of high level induced residual stress on an SU-8 spring fabricated for a MEMS micro-mirror device [3].

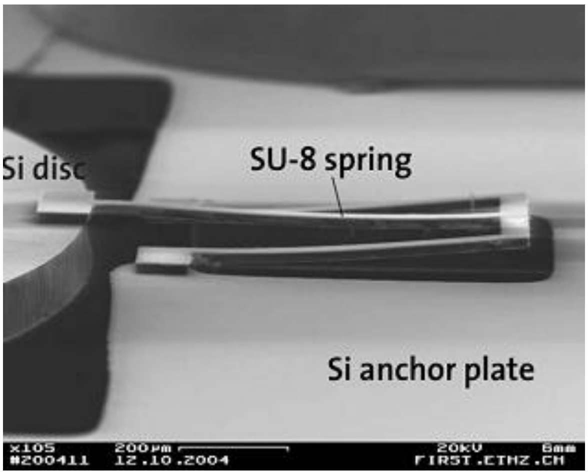

Figure 1. Stress gradient observed in a spring made out of SU-8 [3]

There is a significant amount of research in the literature devoted to understand main factors that affect residual stress. Various techniques are recommended in order to remedy the problem and optimize processing of SU-8 films [3-5]. This paper presents the results of a designed experiment in order to explore the main effects throughout a standard processing of SU-8 films. The type of SU-8 photoresist used in this study is SU-8 2010 from MicroChem® Inc. and seven main factors are examined based on the recommended processing steps and conditions included on the manufacturer's datasheet [6]. The rest of the paper is organized as follows: theory regarding characterization of residual stress and information on material properties of the negative SU-8 photoresist is given. Next, the deposition process and experimental design approach are presented. Finally, obtained statistical analysis and results are presented together with the author's conclusions.

2. THEORY

SU-8 photoresist is an epoxy-based negative tone photoresist (PR) that was introduced in the early nineties and suitable for fabrication of thick layers of structural materials for MEMS devices. The PR is composed of three main components similar to any other PR materials. These include: photo acid generator (PAG), main polymer compound and a solvent. The PAG used in SU-8 material is triarylsulfoniumhexafluroantimonate. This material is responsible for generating acid when PR is exposed to UV-light. The cationic reaction generated by PAG starts polymerization or cross-linking process between highly functionalized 8 epoxy groups of PR's main polymer compound called EPON SU-8. The solvent compound, -Butyrolacton, is responsible for making PR less viscous for better spin coating. It also helps with diffusion of the acid generated by PAG and increase performance of the polymerization process.

The residual stress induced in SU-8 films is a combination of two types of stress categorized as *intrinsic* and *extrinsic* stresses [4]. Intrinsic stress is mostly generated during the aforementioned crosslinking process which is a confinement of monomers in the polymer matrix. It is also stated that loss of mass due to the evaporation of solvent might result in intrinsic stresses. In addition to this, extrinsic stress involves the stress induced due to the coefficient of thermal expansion (CTE) mismatch between Si substrate and SU-8 material. It is formulized as given in eq. 1.

(1)

in this equation, represents the CTE of material, is the induced thermal stress, is Young's modulus, is post-exposure-bake (PEB) temperature and is the ambient temperature. Note that, the higher the difference between PEB temperature and ambient temperature is the higher the induced thermal stress on the film.

The induced stress can be measured utilizing measurement of radius of curvatures of the processed wafers both before and after the deposition of the stress inducing material [7]. The wafer curvature is extracted from a surface profile data obtained by the help of a surface metrology tool such as profilometer. Due to the initial intrinsic curvature of the wafer, two measurements are required, one with and the other one without the stress-inducing film deposition process. Based on the obtained bow height of the wafer from a profilometer tool, the Stoney's equation given in eq. 2 below can be used in order to determine the level of total residual stress in thin film [8].

(2)

in this equation, is stress in the film after deposition, and are substrate radius of curvature before and after deposition. *E* and are Young's modulus and Poisson's ratio of the substrate, respectively. The film thickness and substrate thicknesses are represented with and , respectively.

The radius of curvature can be extracted from the following equation utilizing the wafer bow height of both film and substrate.

(3)

where *H* represents the wafer bow height and R is the radius of curvature.

3. DESIGN OF THE EXPERIMENT

A fractional factorial experimental design is selected in order to obtain a screening experiment and decide which factors are significant on the residual stress induced in SU-8 films through a standard SU-8 PR processing. Seven main effects are included with factor levels given in Table 1. The factors and levels are selected based on the manufacturer's recommended settings for a target film thickness between 6 -15 µm [6]. While selecting the low and high levels, adequate room for *alpha star* points is intended to be left which might be useful for a Central Composite Design approach [9].

Table 1. Main factors selected to be examined in the designed experiment and their levels

Factor Name	**Low Level**	**High Level**
Hard bake temperature	175 ^{o}C	225 ^{o}C
PEB temperature	90 ^{o}C	95 ^{o}C
Dose	110 mJ/cm^2	140 mJ/cm^2
RPM Speed	1500 rpm	3500 rpm
Hard Bake time	10 mins	20 mins
PEB Time	3 mins	4 mins
PAB Time	2 mins	3 mins

Based on the seven factors listed in Table 1, fractionalization element of p=3 is selected to scale the number of treatment combinations (tcs) to 16. This has been done in order to obtain an efficient experimental design that will have the least amount of expenditure on the resources which was the critical number of clean wafers available during the time of the experiment. Additional three center points are added to the design matrix to obtain a better estimate of the residual error during the experiment. The detailed design matrix that includes a total of 19 tcs is given in Table 2. The design matrix includes randomly selected run orders in the first column as well.

In order to select the confounding pattern wisely and minimally violate the confounding rules, a design matrix template given in [9] for seven factors in 16 runs is utilized. According to this design, a factor mapping of alphabet letters from *A* to *G*, the generators become: "$E \approx ABC$, $F \approx BCD$, $G \approx ACD$" and the defining contrast is "$1 \approx ABCE, BCDF, ACDG, ADEF, BDEG, ABFG, CEFG$". This gives the designer a resolution of *IV* which means a confounding of all single effects with the three effect interactions. Note that, three-effect interactions are known to be less likely to occur in most of the experiments. In addition to that, if factors *A* and *B* are found to be not interacting, then *DG*, *DF*, *DE*, and *CD* will be free of confounding as it can be seen from the confounding pattern given in Table 3.

Table 2. Design Matrix of the experiment that includes the whole treatment combinations, settings and randomly selected run orders

Run Order	**TC**	**A: HB Temp [ºC]**	**B: PEB Temp [ºC]**	**C: Dose [mJ/cm^2]**	**D: RPM**	**E: HB Time [minutes]**	**F: PEB Time [minutes]**	**G: PAB Time [minutes]**
19	0	200	95	125	2750	15	3.5	2.5
8	ab(fg)	225	100	110	2500	10	4	3
3	d(fg)	175	90	110	3500	10	4	3
9	b(ef)	175	100	110	2500	20	4	2
5	a(eg)	225	90	110	2500	20	3	3
1	bd(eg)	175	100	110	3500	20	3	3
13	bc(g)	175	100	140	2500	10	3	3
11	c(efg)	175	90	140	2500	20	4	3
7	cd(e)	175	90	140	3500	20	3	2
4	bcd(f)	175	100	140	3500	10	4	2
15	ad(ef)	225	90	110	3500	20	4	2
2	abc(e)	225	100	140	2500	20	3	2
6	-1	175	90	110	2500	10	3	2
16	0	200	95	125	2750	15	3.5	2.5
17	acd(g)	225	90	140	3500	10	3	3
10	ac(f)	225	90	140	2500	10	4	2
18	0	200	95	125	2750	15	3.5	2.5
19	abcd(efg)	225	100	140	3500	20	4	3
14	abd	225	100	110	3500	10	3	2

HB Temp and *PEB Temp* are associated with factors *A* and *B* in the design template due to reason that it is assumed to be no interaction exists between *HB Temp* and *PEB Temp* factors. Thus, the aforementioned two-factor interactions will be left free of confounding by the design itself.

Table 3. Table for confounding pattern

Interaction	**Confounding**
AB ≈	CE, FG
AC ≈	BE, DG
AD ≈	EF, CG
AE ≈	DF, BC
AF ≈	DE, BG
AG ≈	BF, CD
BD ≈	CF, EG

4. PHOTORESIST DEPOSITION PROCESS

Before deposition of the wafers, bare wafer surface profile is extracted for each wafers using Tencor Profilometer and the data is saved into a file with the wafer's unique ID number. A total of 19 wafers are deposited using the settings previously determined and given in Table 2. SCS manual resist coater is used with a modified recipe including two ramped levels for rpm speeds. The first level included a short term intermediate step before the target rpm speed is reached and the second level is equal to the desired target rpm settings in the design matrix. Following the spin coating phase, post application bake is applied using a hot plate. The post application bake is performed at 95 ^{o}C constant temperature for all of the treatment combinations. The PR is flood exposed with *i*-line filter utilizing Karl Suss MA55 Contact Aligner as the radiation source. Before exposing the wafers, in order to adjust the time of exposure to obtain the targeted dose of exposure, intensity of UV-light is measured. Based on the dose formula and the settings in the design matrix, time of exposure is extracted. Right after the exposure step, the wafers are carried near to the hot plates in order to perform post exposure bake step. After PEB, the wafers are developed by using *RER600* edge bead removal which includes PGMEA, chemically. The wafers are developed in a Pyrex dish by adding adequate amount of developer that is enough to cover the whole wafer. After leaving the wafers dipped in the developer approximately 2-3 minutes long, the wafers are rinsed with IPA, DI water and finally dried. The deposited wafers' surface profiles are extracted using the profilometer and bow height information is saved for both before and after the hard bake step.

After all the wafers are processed, residual stress values induced in the SU-8 films are extracted and statistical analysis is performed. The next section provides further information on the analysis and obtained results.

5. RESULT AND ANALYSIS

Equations 1-3 are used in order to calculate the induced residual stress on the processed wafers. The default values used for silicon wafers are: E= 130 GPa, = 0.279 and t_s= 650 µm. The calculated stress levels before and after the hard baking process are given in Table 4. The third column includes the stress levels before hard bake and the last column is the stress levels after the hard bake step.

Table 4. Table for confounding pattern

Run Order	**TC**	**DEV Stress [MPa]**	**HB Stress [MPa]**
19	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	-12.97	-24.20
17	acd(g)	-6.13	-17.65
10	ac(f)	-7.75	-20.66
18	0	-6.90	-17.26
19	abcd(efg)	-9.41	-21.39
14	abd	-6.40	-16.71

Statistical analysis is performed utilizing JMP software and the response surface is generated utilizing the obtained information. Figure 1 below shows the results of ANOVA analysis after the insignificant effects are excluded from the model. According to ANOVA analysis, the obtained model is statistically significant when a 5 % alpha risk is considered.

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	17.117675	4.27942	11.7880
Error	13	4.719419	0.36303	Prob > F
C. Total	17	21.837094		0.0003

Figure 2. ANOVA results obtained by the statistical analysis

Figure 2 below includes the parameter estimates for the model equation and showing the most significant factors that play important roles in the residual stress for SU-8 film when an alpha risk level of 5 % is considered. According to these results: *Dose*, *PEB Time* as single effects and *PEB Temp*Dose* and *Dose*PEB Time* 2-effect interactions are the most significant factors.

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-6.999444	0.142016	-49.29	<.0001
Dose	-0.793125	0.15063	-5.27	0.0002
PEB Time	-0.424375	0.15063	-2.82	0.0145
PEB Temp*Dose	-0.378125	0.15063	-2.51	0.0261
Dose*PEB Time	-0.343125	0.15063	-2.28	0.0403

Figure 2. Parameter estimates for model equation showing the most significant factors

Based on the parameter estimates given in Figure 2, the model equation, in design units, can be written such as given in eq. 4. In this equation, represents the estimated level of residual stress based on the model.

(4)

Note that, the optimum stress level on the film is expected to be zero which represents no stress. The negative stress levels represent compressive type of residual stresses induced. According to this model, the optimum settings can be selected as follows: *Dose*= 110 mj/cm^2 , *PEB Time*= 3 mins, *PEB Temp*= 95 ^{o}C which gives an estimate of -5.738 MPa compressive stress.

Figure 3 below shows the leverage plots obtained for the significant factors. These plots show how the response variable changes over the significant factors statistically identified with the above results. These plots show that increasing the *Dose* and *PEB Time* also increase the residual stress level induced in SU-8 films.

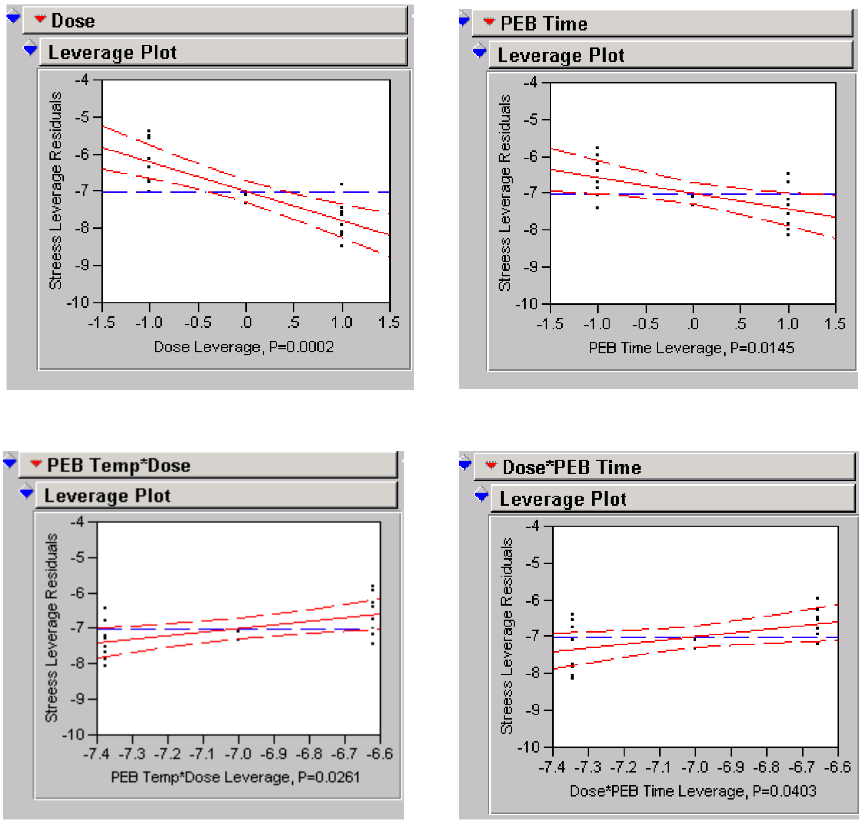

Figure 3. Leverage plots showing how the response variable changes versus the level of significant factors

6. DISCUSSIONS

The obtained results presented in the previous section match with the similar research work performed by other researchers in the literature [4, 5]. In [4] Keller *et al.* also state that *Dose*, *PEB-Time* and *PEB temperature* highly affect the residual stress induced in SU-8 thin films. In addition to this, they also include the solvent content in the resist as a significant factor which is regulated by the *PAB time* or *evaporation time* before exposure. The reason is stated as: higher solvent concentration facilitates distribution of photo acid, generated by PAG compound in the PR. By this means, the amount of polymerization or cross-linking is increased, so that the residual stress. According to the results obtained in our experiment, *PAB Time* does not have any significance on the residual stress. This might be due to several reasons: it might be possible that the upper and lower limits of *PAB Time* are not large enough in order to observe the difference on the levels of the residual stress. In addition to that, the amount of solvent left inside the PR bottle used in this experiment might not be at an adequate level due to the expiration date of the SU-8 resist. The difficulties experienced with spin coating of the PR, when the exact recommended settings and information on spin-speed curves are followed, also support this point of view.

When the residual stress levels after the hard bake process is taken into account, all of the factors become statistically insignificant. This might be due to the reason that hard bake process introduces a lot of noise to the results since the hard bake temperature levels are beyond the glass transition temperature of the polymer material and a large/irrelevant variation might be induced on the response variable.

7. CONCLUSIONS AND FUTURE WORK

A thorough understanding of SU-8 PR processing and residual stress characterization is gained through the experimental investigation of residual stress in SU-8 films. The obtained results are statistically significant within reasonable alpha risk levels and are in good agreement with the previously published work by the other researchers in the literature. *Dose*, *PEB Time* and *PEB Temp*Dose*, *Dose*PEB Time* interactions are found to be significant. When the nature of the cross-linking process is considered, the significant factors are directly related to the phenomena that controls the stress levels.

Possible future work might include running several alpha star points as in a central composite design approach and increase the number of levels for the factors to a level greater than two. By this way, the non-linear interactions might also be possible to obtain. In order to investigate the other significant interaction effects confounded with the effects included in the model, the deconfounding techniques can also be implemented. Finally, additional wafers can be processed with the optimum settings and the accuracy of the model might be tested in a real case.

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