Characterization of Negative Photoresist Processing by Statistical Design of Experiment (DOE)

Sei-Young Mun, Gwang-Beom Kim, Dea-Wha Soh, and Sang Jeen Hong, *Member, KIMICS*

Abstract—SU-8 is a epoxy based photoresist designed for MEMS applications, where a thick, chemically and thermally stable image are desired. However SU-8 has proven to be very sensitive to variation in processing variables and hence difficult to use in the fabrication of useful structures. In this paper, negative SU-8 photoresist processed has been characterized in terms of delamination, based on a full factorial designed experiment. Employing the design of experiment (DOE), a process parameter is established, and analyzing of full factorial design is generated to investigate degree of delamination associated with three process parameters: post exposure bake (PEB) temperature, PEB time, and exposure energy. These results identify acceptable ranges of the three process variables to avoid delamination of SU-8 film, which in turn might lead to potential defects in MEMS device fabrication.

Index Terms—SU-8, Post Exposure Bake (PEB), Statistical Design of Experiment (DOE)

I. INTRODUCTION

SU-8 is a high contrast, epoxy based photoresist designed for micromachining and other microelectronic applications, where a thick, chemically and thermally stable image are desired. It is non-conductivity and so can be used to a dielectric in electroplating. Thus SU-8 potentially has many advantages of other thick film techniques, and few of their specific disadvantages. For these reasons, it has been widely used by MEMS devices.

Despite the advantages of SU-8, Thick layers of SU-8 experienced more stress, and the structures tended to delaminate more quickly than thin layers [1]. SU-8 has proven to be very sensitive to variation in processing variables and hence difficult to use in the fabrication of useful structures. The literature describes wide variation in values for processing parameters which reflects the different SU-8 recipes used [2]. Therefore, this paper investigates the variation of SU-8 processing, with the ultimate goal of minimizing delamination, using statistical design of experiment (DOE).

II. EXPERIMENT

A. Statistical designed experiment

The traditional method of collecting large quantities of data by holding each factor constant in turn until all possibilities have been tested is an approach that quickly becomes impossible as the number of factors increases. Statistical experimental design is a systematic, efficient, and alternative methodology for characterization and modeling using a relatively small number of experiments [3].

Full factorial experiments are suitable when there are many factors of interest in an experiment. The perfect experimental design is a full factorial, with replications, that is conducted in a random manner. The number of treatment conditions is determined by:

$$TC = k^n \quad (1)$$

Where TC is the number of treatment conditions, k is the number of levels, and n is the number of factors. However, a good experiment must be efficient. If the experiment is conducted on production equipment with long setup times, full factorial design may be in efficient [4].

In this paper, three input factors with three levels of each were considered: Exposure energy, post exposure baking (PEB) temperature, and time. Since cross-linking takes place after exposure, the variables in the soft baking step were later omitted. Instead, the soft baking step was performed in a consistent manner for all samples.

B. Fabrication

SU-8 was spin coated on 4 inch silicon wafers to a thickness of 100μm, and the samples were soft baked at 70℃ on a hot plate to drive off solvents. Based on the designed experiment, all possible orthogonal combinations of three parameters were applied. All samples were developed for a fixed time, and the degree of delamination was measured. The degree of delamination was quantified by the following expression:

$$\text{Delamination} = \frac{(L-l)}{L} \times 100(\%) \quad (2)$$

where L is the length of the original bar pattern, and l is the length of bar pattern that remained on substrate (see Figure 1). To minimize measured error, l was averaged over eight bar patterns in one location.

Manuscript received November 20, 2005.

Sei-Young Mun is a senior student in the Department of Electronic Engineering at Myongji University.

Gwang-Beom Kim is a senior student in the Department of Electronic Engineering at Myongji University.

Dae-Wha Soh is a professor in the Department of Electronic Engineering and director of semiconductor technology center at Myongji University.

Sang Jeen Hong is a with the Department of Electronic Engineering & NBRC at Myongji University, 38-2 San, Nam-dong, Yongin, Gyeonggi-do, Korea, 449-728 (Tel: +82-31-335-6374, Email: samhong@mju.ac.kr)

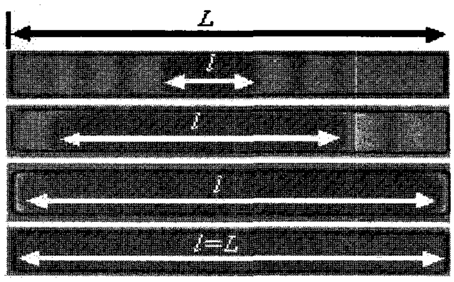


Fig. 1 Different degrees of delamination

III. RESULT AND DISCUSSION

Once a series of experiment for investigating the delamination, the amount of delamination was measured from each sample, and a statistical regression model for delamination was established followed by the suggested order provided from the *Minitab* (commercial statistical analytic software). To investigate the fitness of the model, first, residual analysis has been performed. Residuals mean how the observed value is far from main trend of data, and they are used for the analysis of variability of the model.

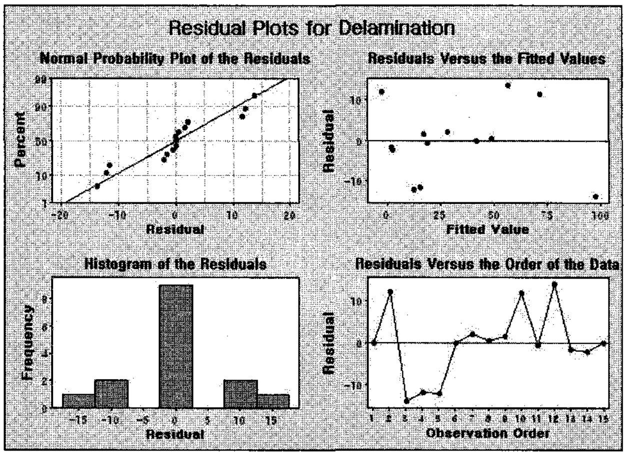


Fig. 2 Residual plots for delamination

According to the normal probability plot provided in Fig. 2, we have confirmed that the data set follows underlying assumption of normal distribution. In addition, other residual plots vs. fitted value and observation order show fairly random distribution. From this result, one can infer that the established model was not biased or violate underlying assumption in basic statistics.

To investigate individual effect of input parameters on delamination, main effect plots were studied in Fig. 3. In the figure, if the line is straight line through out the parameter window, one can infer that the corresponding parameter does not affect on the response; however, we can confirm that all the parameters at different levels are different from each other, and this shows significant main effect of all three input parameters on the delamination.

Traditionally, experimenters tend to investigate their experimental parameters by fixing other associating parameters at certain values under the assumption that those parameters are independent and do not interact each other.

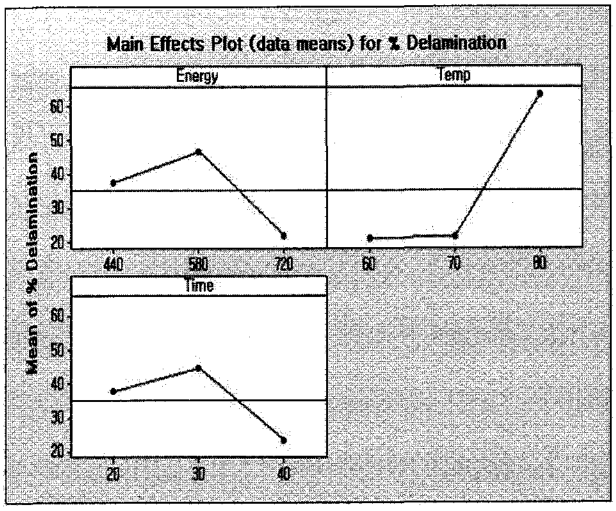


Fig. 3 Main effect plots for delamination

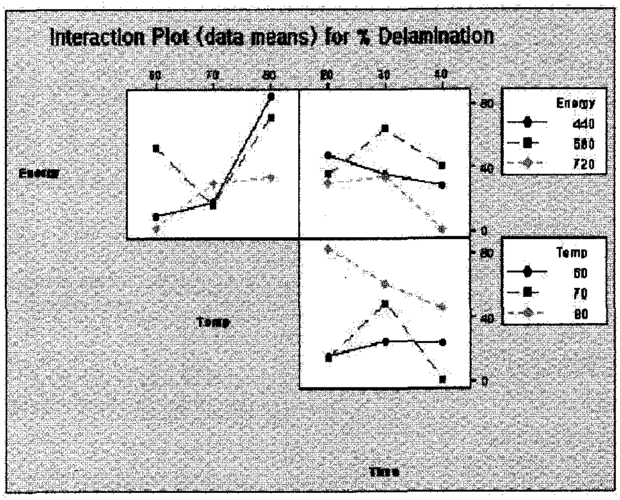


Fig. 4 Interaction plots for delamination

This is not true for most of the case, especially in this type of experiment. Therefore, we also have investigated interaction between parameters. If there is any interaction between two or more parameters, then the interaction must be considered in the statistical model to increase model accuracy. As shown in Fig. 4, any two parallel lines present no interaction between two parameters. For instance energy and temperature has very little interaction between 30-40% of delamination range, and this interaction can be neglected in this statistical model. However, most of the case, the interaction between parameters associated with measured delamination cannot be neglected, and this should be included in the model for higher accuracy and model credibility. In this sense, we have included two interaction terms in the model and they are listed in Table 1.

The analysis of variance (ANOVA) table derived from the established model present statistical model accuracy. Main effects and interaction terms included model were statistically tested by (F-test), and their individual p-value (the probability that the term falls beyond the confidence level, in this case confidence level α=0.90) is presented. According to the p-value of the interaction between PEB temperature and PEB time, it slightly slipped out statistically not significant region; however, it was not omitted in the model since it was regarded somewhat significant factors in negative photoresist processing based on previous experience. In this sense, PEB time was also included in the model considering interaction as well.

Table 1. ANOVA table for the general linear model

Source	DF	Seq SS	Adj MS	F	P
Exposure energy	2	2881.3	1440.6	2.96	0.090
PEB temperature	2	10688.2	5344.1	1098	0.002
PEB time	2	2153.6	1076.8	2.21	0.152
Exposure energy *PEB temperature	4	6154.0	1538.5	3.16	0.054
PEB temperature *PEB time	4	3869.5	967.4	1.99	0.161
Error	12	5869.5	486.8		
Total	26	31588.5			

It is observed that the higher the temperature, the large the degree of delamination. In addition, PEB time also somewhat affects the degree of delamination at a given temperature. The shorter the PEB time, the less cross-linking, increasing the degree of delamination after a certain temperature. The high degree of delamination over 720℃ is primarily due to the coefficient of thermal expansion (CTE) mismatch between SU-8 and the silicon wafer with native oxidation. CTE mismatch can be explained in the following equation.

$$\sigma_{th} = (\alpha_s \alpha_f)\Delta T\left(\frac{E}{1-v}\right) \tag{3}$$

α_s and α_f are the coefficient of thermal expansion of substrate and film, ΔT is the temperature range, E is Young's modulus of substrate and v is Poisson's ratio of substrate. We can know effect that three factors get to delamination through contour plots provided in Fig. 5-7.

It is observed that the higher the temperature, the large the degree of delamination. In addition, PEB time also somewhat affects the degree of delamination at a given temperature. The shorter the PEB time, the less cross-linking, increasing the degree of delamination after a certain temperature. The high degree of delamination at temperatures above 70 ℃ is primarily due to the coefficient of thermal expansion (CTE) mismatch between SU-8 and the silicon wafer with native oxidation. As the exposure energy increases, the degree of delamination decreases, while the temperature at which the delamination starts to occur decreases. Lower exposure energy tends to decrease cross-linking of the polymer in the exposed area, and consequently, this decreases adhesion. Lake of resist adhesion leads to distorted patterns on the wafer surface. The PEB time is less than 30 minutes and the exposure energy is less than 580 mJ/cm^2, showed some degree of delamination due to incomplete cross-linking.

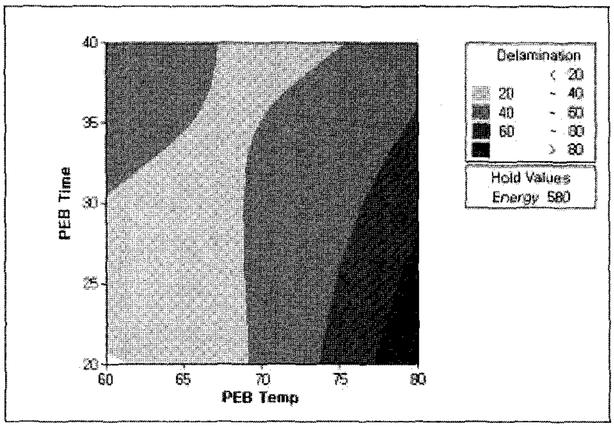


Fig. 5 Countour plot (Energy 580 mJ/cm²)

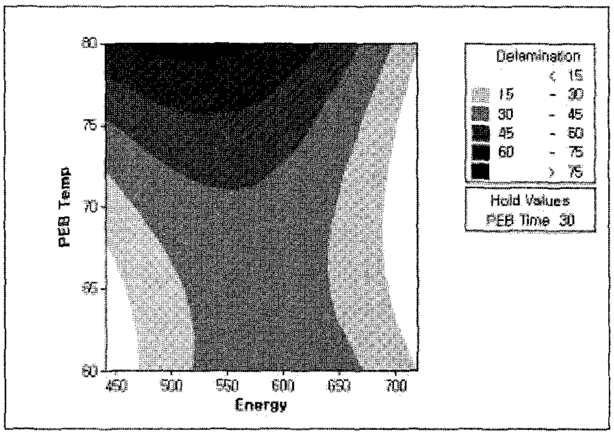


Fig. 6 Countour plot (PEB time of 30min)

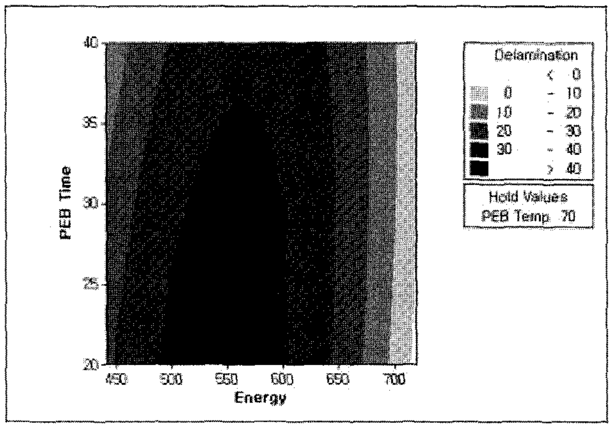


Fig. 7 Contour plot (PEB temp of 70℃)

IV. CONCLUSION

In this paper, three significant parameters associated with SU-8 delamination were investigated, and their effects on delamination determined found. Higher PEB temperatures at a fixed PEB time result in more delamination due to CTE mismatch. In addition, the greater dose of exposure energy at which cross-linking occurs, the less degree of delamination. These results identify acceptable ranges of the three process variables to avoid delamination of SU-8 film, which in turn might lead to potential defects in MEMS device fabrication.

REFERENCES

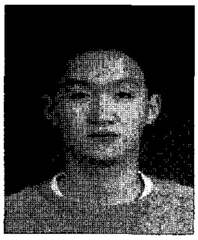
[1] M. Brunet, T. O'Donnell, J. O'Brien, P. McCloskey, and S. Mathuna, "Thick photoresist development for the fabrication of high aspect ratio magnetic coils," *J. Micromech. Microeng.*, vol. 12, 2002, pp. 444-449.

[2] M. Despont, H. Lorenz, N. Fahrni, J. Brugger, P. Renaud, and P. Vettiger, "High-aspect ratio ultrathick, negative tone near-UV photoresist for MSMS applications," *In Proc. IEEE, 10th Anuual Int. Workshop in Micro Eletro Mechanical Systems*, Nagoya, Japan, pp. 518-522, Jan. 1997.

[3] G. Box, W. Hunter, and J. Hunter, *Statistics for Experimenters*, New York: Wiley, 1978.

[4] Besterfield, D., Besterfield, C., Besterfield, G. H., and Besterfield, M., *Total quality development*, Upper Saddle River, NJ: Prentice Hall, 1999.

[5] Robert H. Carver, "*Doing Data Analysis with MINITAB 14*," Brooks Cole., 2003.



Sei-Young Mun
He is an undergraduate research student of Intelligent Micro/Nano Process Lab. at Myongji University, Yongin, Korea.
His research interest is statistical process control (SPC) and Semiconductor manufacturing technology.

Kwang-Beom Kim
He is an undergraduate research student of Intelligent Micro/Nano Process Lab. at Myongji University, Yongin, Korea.
His research interest is advanced process control (APC) and Semiconductor manufacturing technology.

Dea-Wha Soh
He received the B.S. degree in electrical engineering from Hanyang University, Korea, in 1972 and the M.S. degree in electrical engineering from Myongji University, Korea, in 1975 and Ph.D. degree in electrical engineering from Kyunghee University, Korea, in 1987. Currently, he is a professor in the Department of Electronic Engineering at Myongji University, Yongin, Korea. His research interest is semiconductor material and device fundamentals.

Sang-Jeen Hong
He received the B.S. degree in electrical engineering from Myongji University, Korea in 1999 and the M.S. and Ph.D. degree in electrical and computer engineering from the Georgia Institute of Technology, Atlanta, in 2001 and 2003, respectively. Currently, he is an assistant professor in the Department of Electronic Engineering at Myongji University, Yongin, Korea, and he also serves as an editorial board of *KIMICS* and *KIEEME*. His research interest is statistical process control (SPC) and advanced process control (APC), real-time fault detection and classification, and plasma induced damage in plasma etch process focused on high volume semiconductor manufacturing environment.