Influence of photo-initiator concentration on residual mechanical stress in SU-8 thin films

Influence of photo-initiator concentration on residual mechanical stress in SU-8 thin films

S.D. Psoma*, D.W.K. Jenkins, G.E. Derbyshire and R. Stevens

Central Microstructure Facility, Engineering and Instrumentation Dept., CCLRC,
Rutherford Appleton Laboratory, Chilton, Didcot, Oxfordshire OX11 0QX, U.K.
* *E-mail: S.Psoma@rl.ac.uk*

ABSTRACT

In this paper, experimental results on the residual mechanical stress generated during the different steps of the photolithography process of SU-8 thin films using different photo-initiator concentrations are presented. The main aim of the reported research work has been firstly to investigate and identify the causes for the generation of residual stresses and secondly to optimise the fabrication process in order to minimise the residual stresses. It was found that the major parameters that influence the generation of internal stresses were the exposure time, the post-baking temperature and time and the concentration of the photo-initiator which is used (Cyracure UVI). At lower concentrations of the photo-initiator, the measured stress levels after the post-baking step were consistently found to be significantly lower than the ones corresponding to higher photo-initiator concentrations. In addition, there was an additional improvement by using a ramping hot-plate. Finally, preliminary experiments were carried out in order to encapsulate nanoparticles in these low stress SU-8 thin films.

Keywords: SU-8 films, residual stress, photo-initiator, fabrication, negative photoresist

1 INTRODUCTION

The negative photoresist SU-8 is widely used in the development and fabrication of micro- and nano-mechanical structures for MEMS applications such as electrostatic sensors and actuators, biosensors, micromolds, microfluidic channels or packing applications [1-4]. The structures of the resist are the outcome of photochemical and thermal cationic processes and result in the formation of vertical sidewalls and high aspect ratio features, which are among SU-8's most desirable attributes [5-7].

There are two types of processes developed for producing high-aspect-ratio microstructures. The first one is the LIGA process that utilizes synchrotron deep X-ray lithography, and it is a very expensive and not widely available technique [6]. The second more commonly used process is a LIGA-like process using UV optical lithography, which has been developed and demonstrated as an economical and effective way to produce high aspect ratio SU-8 microstructures [2-4]. However, during the fabrication process of SU-8 structures or films, considerable internal stress is generated, which causes the appearance of undesirable cracked features which consists its main drawback. This is due to its very high coefficient of thermal expansion (CTE) at around 50 ppm/K, in comparison to commonly used substrates such as silicon (2.3 ppm/K for <111> and 1.8 ppm/K for <100>) or glass (around 10 ppm/K). During the process steps this internal residual stress is likely to rise to unacceptable levels. In the literature the most commonly utilized tool for investigating this aspect is the computational process simulation based on Finite Element Analysis (FEA) [7, 9].

The aim of this work is the reduction and control of the internal stress through the modification of the chemical composition of the SU-8 resist, without deterioration of the lithographic properties of the material as compared to conventional SU-8. For this reason, an experimental investigation was carried out in which the influence of different concentrations of a photo-initiator on the residual stress was studied.

2 EXPERIMENTAL

The SU-8 resist was prepared by dissolving the resist Epon SU-8 (Epikote 157) in an organic solvent, the γ-butyrolactone (GBL, Avocado Research Chem. Ltd), and by adding the curable photo-initiator UV1-6992 (DOW Chem. Co.). The quantity of the solvent determines the viscosity and thereby the range of available thicknesses. In all experiments, the composition of 40/60 SU-8 / solvent with different percentages of UV1-6992 was used as can be seen in Table 1. The final product was left in the roller mixer for 96 hours and also to mature for at least two weeks.

All the 4 inch silicon wafers <100> used in the present study were firstly cleaned for residual organic in an oxygen plasma, baked at 90 ºC for 5 min, and then allowed to cool down. At this stage, the reference scan of the wafer for the stress measurements was taken using the profile-meter. Resist application was performed using a standard spinner (Karl Suss Gyrset) and constant dispense volume for a given thickness and subsequently was allowed to relax for 15 min before baking. The soft-baking was at 75 ºC for a duration of 10 min in a hot plate. Subsequently the resist film was exposed with a standard UV aligner (Karl Suss MA 6), and finally the crosslinked SU-8 was post-baked in a hot plate for 5 min.

SU-8 Resist	SU-8 granules	GBL solvent	UV1 photo-initiator
40/60 1%	40 gr	59 gr	1 gr
40/60 2.5%	40 gr	57.5 gr	2.5 gr
40/60 5%	40 gr	55 gr	5 gr
40/60 7.5%	40 gr	52.5 gr	7.5 gr
40/60 10%	40 gr	50 gr	10 gr

Table 1: Chemical compositions for preparing SU-8 resists with different percentages of photo-initiator.

In all process steps, stress measurements were performed because stress was generated in the SU-8 films and wafers as a result of a thin film deposition from the outset of the experiments. The deformation of the thin film generated bending and compressing (compressive stress) or expansion (tensile stress) of the substrate surface, which is silicon wafer <100> in the present study. Careful monitoring of the thin film stress data was useful for reducing process variations.

The average stress was calculated according to the Stoney equation (1) for stress in a thin-film layer deposited on a substrate, which is as follows:

$$\sigma = \frac{1}{6R}\frac{E}{(1-v)}\frac{t_s^2}{t_f} \qquad (1)$$

where

$$\frac{E}{(1-v)} = \text{wafer elastic constant}$$

σ = stress
t_s = wafer thickness
t_f = film thickness
R = radius of curvature
E = Young's Modulus for the wafer (substrate)
V = Poisson's Ratio

The KLA-Tencor P-15 Profiler software was used for calculating the average stress values for the samples. In addition, the NewView 200 software for film applications of the interferometer Zygo (Zygo Corp.) was used for the surface topology images of the SU-8 films.

3 RESULTS AND DISCUSSIONS

After spinning the SU-8 thin films (from 1 µm to 15µm) on the wafers, the effect of the soft-bake on stress was investigated. Different temperatures were used from 60 ºC to 160 ºC and also for different durations from 2 min to 30 min. Despite the high temperatures (up to 160 ºC) and the long duration (up to 30 min), only a small compressive stress was introduced in the range of 1 to 3.5 MPa for the film thickness of 1 µm to 15 µm on the wafer. The appearance of the stress is because of the difference between the thermal expansion coefficients (TECs) of the wafer and the SU-8 films. However, it is relatively low because during the cooling phase of the soft-bake, polymer chain rearrangements take place in the resists that are not crosslinked. For this reason, in all experiments of the present study soft-bake at 75 ºC was used for 10 minutes.

The main stress was generated as the crosslinked SU-8 cooled down. Figure 1 shows the variation of the measured stress as a function of the exposure time for four different thicknesses of the 40/60 SU-8 that are equal to 1.2, 4, 7 and 11µm. In this case, the concentration of the photo-initiator UV1 was set equal to 10%. A fast growth of the compressive stress level appears for all cases with the increase of the exposure time. At a given exposure time, the stress is found to be highest for the 1.2µm film and reduces with the increase in thickness.

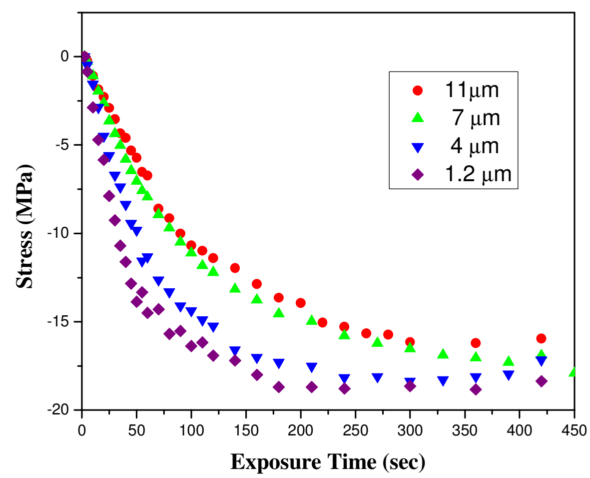


Figure 1: Variations of the measured stress as a function of the exposure time for different thickness of 40/60 SU-8 with 10% photo-initiator UV1.

Similar trends in the dependence of the residual stress on exposure time have been produced when using a 5% concentration of the photo-initiator. The experimental results from this investigation are presented in Figure 2 where four different thicknesses of the SU-8 film, equal to 1.4, 6.2, 8.3 and 11µm, were considered. The increase of the compressive stress with exposure time is apparent and in addition the thickness variation has a considerable effect on stress levels.

Figure 3 presents the variation of the measured stress as a function of the exposure time for three different concentrations of the photo-initiator. The concentration of the photo-initiator appears to have a significant influence in the SU-8 thin films and its reduction from 10% to 1% is associated with a significant drop in the level of the residual stresses. However, by reducing this concentration, the exposure time has to be increased in order to accomplish the crosslinking of the resist. It was found for a good lithography it must be used SU-8 resist with at least 2.5% photo-initiator.

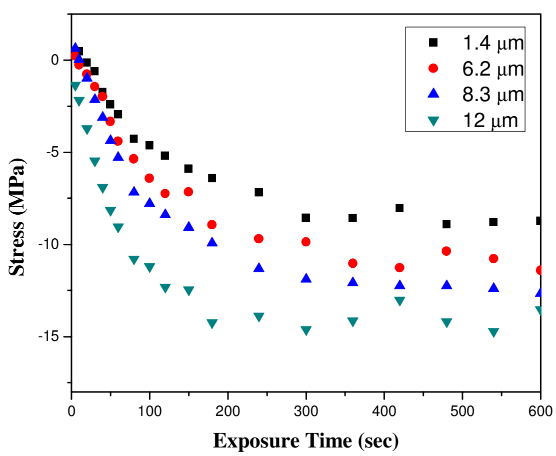


Figure 2: Variations of the measured stress as a function of the exposure time for different thickness of 40/60 SU-8 with 5% photoinitiator UV1.

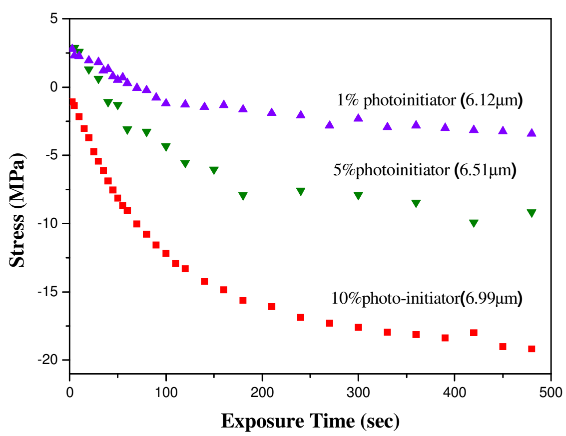
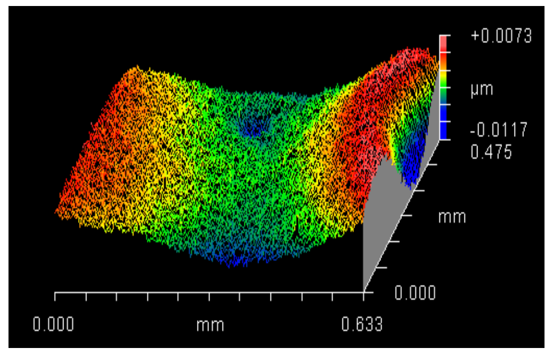
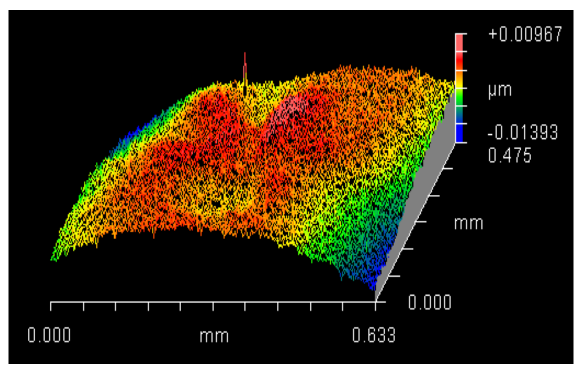


Figure 3: Variations of the measured stress as a function of the exposure time for three different concentrations photo-initiator UV1 of the 40/60 SU-8 resist.

Figure 4a illustrates the surface topology of the SU-8 film with 2.5% photo-initiator after the soft baking process. As it can be observed, there is a deepening in the middle section and this is associated with a tensile stress at a low level (approximately 2MPa). In Figure 4b, the morphology of the SU-8 film surface exhibits an opposite behaviour and the existence of a compressive stress after an exposure time of 40 sec. The stress levels increase significantly after the post-baking phase and they change rapidly becoming tensile (approximately 25MPa).



(a)



(b)

Figure 4: Surface topology of the 40/60 SU-8 film with 2.5% photo-initiator (a) after the softbake at 75 ºC (compressive stress) and (b) after the exposure time of 40 sec (tensile stress).

The influence of the duration of the post-baking process on the measured residual stress is illustrated in Figure 5 where in all cases a tensile stress has been found to be present. The concentration of the photo-initiator has a significant influence on the stress levels and an increase in this concentration causes a marked increase in stress levels.

The stress results of the different post-baking temperatures were processed in such a way that all curves start from the same initial value of 0 for the stress.

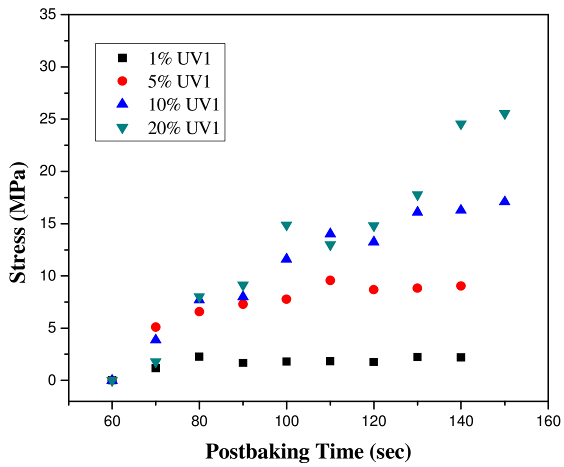


Figure 5: Variations of the measured stress as a function of the postbaking temperature for four different concentrations of the photo-initiator UV1 of the 40/60 SU-8 resist.

It was found that using a ramping hot plate during the postbaking step, the tensile stress was slightly reduced. This was significantly improved by adding relaxation times between all process steps.

Finally, preliminary experiments were carried out in order to investigate the stress effect of SU-8 thin films with entrapped nanoparticles using different concentrations of the photo-initiator. It was found that the new uniform SU-8 nanocomposite material/film had reduced stress levels, as compared to the pure SU-8 film, by a factor of 3 when using 5% and 2.5% of photo-initiator UV1.

4 CONCLUSIONS

Internal residual stress occurs in negative tone photoresist SU-8 caused by the difference between the coefficients of thermal expansion of the resist layer and the substrate. The network density of the exposed and postbaked photoresist also influences the value of stress. Stress reduction can be attained by optimizing the process parameters as well as by a modification of the chemical composition of the photoresist. This stress can be significantly reduced by lowering the concentration of the photo-initiator both during the exposure and the post-baking process. The minimum photo-initiator concentration must be at least 2.5% in order to accomplish proper lithography. In addition, during the process steps of the softbaking and the postbaking a slow warming up and also cooling down procedure using a ramping hot plate, with long relaxation times after post exposure bake and maybe special mask design can be implemented in order to improve the stress behavior of the photoresist. Finally, low stress thin films of SU-8 can be used for encapsulating nanoparticles or to improve the biocompatibility of materials used in the field of biosensors. In addition, the new low stress SU-8 nanocomposite films can be utilized in coating for moving parts or micro-nano structures and also in other MEMS applications.

REFERENCES

[1] K.Y. Lee, N. LaBianca, S.A. Rishton, S. Zolgharnain, J.D. Gelorme, J. Shaw and T.H.-P. Chang, "Micromachining applications of a high resolution ultrathick photoresist", J. Vac. Sci. Technol. B, Vol. **13**(6), pp 3012-3016, 1995.

[2] H. Lorenz, M. Despont, N. Fahrni, N. LaBianca, P. Renaud and P. Vettiger, "SU-8: a low-cost negative resist for MEMS", J. Micromech. Microeng. 1997, Vol. 7, pp 121-124.

[3] H. Lorenz, M. Despont, N. Fahrni, J. Brugger, P. Vettiger and P. Renaud, "High-aspect-ratio, ultrathick, negative-tone near-UV photoresist and its applications for MEMS", Sensors and Actuators A, Vol. 64, pp 33-39, 1998.

[4] D. Esinenco, S.D. Psoma, M.Kusko, A. Schneider and R. Muller, "SU-8 micro-biosensor based on Mach-Zehnder interferometer", Rev. Adv. Mater. Sci., Vol. 10, pp 295-299, 2005.

[5] C.G. Khan Malek, "SU8 resist for low-cost X-ray patterning of high-resolution, high-aspect-ratio MEMS", Microelectronics Journal, Vol. 33, pp 101-105, 2002.

[6] H. Lorenz, M. Laudon and P. Renaud, "Mechanical characterization of a new high-aspect-ratio near UV-photoresist", Microelectronic Engineering, Vol. 41/42, pp 371-374, 1998.

[7] M.C. Peterman, P. Huie, D.M. Bloom and H.A. Fishman, "Building thick photoresist structures from the bottom up", J. Micromech. Microeng., Vol.13, pp 380-382, 2003.

[8] E.W. Becker, W. Ehrfeld and P. Hagman, "Fabrication of microstructures with extreme structural heights by synchrotron radiation lithography, galvanoforming and plastic forming (LIGA process), Microelectronic Engineering, Vol. 4, pp 35-56, 1986.

[9] B. Li, M. Liu and Q. Chen "Low stress ultra-thick SU-8 UV photolithography process for MEMS"; J. Microlith., Microfab., Microsyst., 2005, Vol. 4 (4), pp43008-43013.