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Effects of temperature on mechanical properties of SU-8 photoresist material †

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Abstract

A representative fabrication processing of SU-8 photoresist, Ultraviolet (UV) lithography is usually composed of spin coat, soft bake, UV exposure, post exposure bake (PEB), development and optional hard bake, etc. The exposed region of SU-8 is crosslinked during the PEB process and its physical properties highly depend on UV exposure and PEB condition. This work was initiated to investigate if thermal baking after fabrication can affect the mechanical properties of SU-8 photoresist material because SU-8 is trying to be used as a structural material for MEMS operated at high temperature. Since a temperature of 95°C is normally recommended for PEB process, elevated temperatures up to 200°C were considered for the optional hard bake process. The viscoelastic material properties were measured by dynamic mechanical analyses (DMA). Also, pulling tests were performed to obtain Young's modulus and Poisson's ratio as a function of strain rate in a wide temperature range. From this study, the effects of temperature on the elastic and viscoelastic material properties of SU-8 were obtained.

Keywords: SU-8; Elastic constants; Visco-elasticity; Thermo-mechanical effects; Dynamic mechanical analysis (DMA)

1. Introduction

SU-8 [1], a negative, epoxy-type and near-UV photoresist material, has been developed for applications requiring high aspect ratios in very thick layers. Since SU-8 has low optical absorption in the near-UV range, it is possible to give rise to vertical sidewall profile with good dimensional control over the entire height. Moreover, the interest of SU-8 in MEMS (micro electro mechanical systems) applications are increasing because SU-8 has the advantage of the capability to self-planarize during prebake and to eliminate the edge-bead effect as well as low cost [2, 3]. Many studies have been performed to develop a new fabrication scheme for better performance such as high resolution and aspect ratio of SU-8 structures [4-10]. For the successful utilization of SU-8 as a structural material in MEMS, it is a prerequisite to characterize mechanical and thermal properties in MEMS scale. Therefore, a mechanical characterization has been conducted through many works. Biaxial modulus of elasticity and the CTE (coefficient of thermal expansion) of SU-8 were determined by measuring warpage of a 20 m thick resist layer on Si substrates subjected to thermal loading from 20°C to 95°C [11]. The shear strength of the cantilevered SU-8 micro-posts fabricated on silicon substrates was measured using the static cantilever beam bending [12]. The depth-sensing micro-indentation test was proposed to obtain Young's modulus and hardness of thin films [13, 14]. Young's modulus of the proton beam exposed SU-8 was determined using a stylus-type load-deflection method [15]. From the aforementioned works, Young's modulus of SU-8 has been investigated [3, 7, 11, 12, 15], where it is about 2.4 ~ 5.5 GPa depending on fabrication and testing conditions. Since the properties of SU-8 are very sensitive to the process parameter variation, some researchers have studied the effect of process conditions for SU-8 fabrication on the material properties or lithographic performance [6, 15-17]. The effects of curing conditions such as thermal baking time, thermal baking temperature and UV exposure dose on the thermal and mechanical properties were evaluated [16]. It was shown that Young's modulus was dependent on the proton beam exposure dose [15]. The influence of soft bake time [6] and temperature [17] on the lithographic performance and cracking was investigated, also.

Currently, the efforts to apply SU-8 to MEMS structure such as micro-fluid channel where hot liquid flows have been carried out, and the material properties at elevated temperatures are necessary for reliability of new micro structures. Although the material properties of SU-8 are highly dependent on temperature due to the characteristics of polymer, the effects of operating temperature on material properties of SU-8 have not been explored precisely. Also, the authors realized it during tensile tests that the mechanical properties of SU-8 after fabrication could be changed according to temperature and temperature holding time (THT). This means that the

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Fig. 1. Layout of samples on the mask.

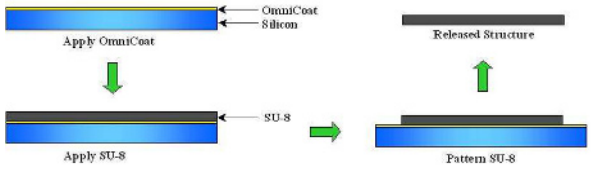


Fig. 2. Process for fabrication of SU-8 samples.

additional thermal curing can affect the mechanical property of SU-8 and it can be changed according to optional hard bake (HB) conditions.

In this study, the elastic material properties of SU-8 were measured as a function of strain rate in a wide range of temperature, from 25°C to 200°C. Also, THT at a specific temperature was varied to systematically investigate the thermal curing effect on SU-8 after fabrication. From the results, it is recommended that the hard bake process is necessary for reliable material properties of SU-8 at elevated temperatures. Since viscoelasticity provides a usable engineering approximation for many applications in polymer and composites engineering [18], dynamic mechanical analyses (DMA) are performed to investigate the optimal hard bake condition.

2. Fabrication of SU-8

Since SU-8 is a negative UV-photoresist [19], wherein the area of the resist exposed to UV light solidifies during development and the area unexposed to UV dissolves in the developer, dog-bone shaped bright openings were created as shown in Fig. 1. The mask was designed for 4 inch diameter silicon substrate. In this study, SU-8-100 produced by MicroChem Corporation was used and dog-bone shaped samples of 35 mm length, 4 mm width and 0.12 ~ 0.15 mm thickness were considered. Fig. 2 shows the fabrication process flow. SU-8 belongs to the epoxy group of polymers that are characterized by excellent adhesion. Moreover, cross-linked SU-8 is known to be extremely difficult to remove. Hence, OmniCoat, an adhesion promoter / release layer was used to reduce adhesion between SU-8 and silicon. OmniCoat was coated on wafer by spinning and dynamic dispense. Dynamic dispense involves dispensing at low spinning speed (generally around 500 rpm) to improve wettability and eliminate voids that may otherwise form. OmniCoat was spun for 5 seconds at 500 rpm with an acceleration of 100 rpm/s and 30 seconds at 3000 rpm with an acceleration of 300 rpm/s. Thickness of OmniCoat was about 17 nm. The wafer was baked for 1 minute at 200°C on a hot-plate and then allowed to cool to room temperature before beginning SU-8 processing.

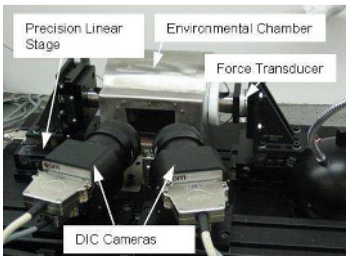


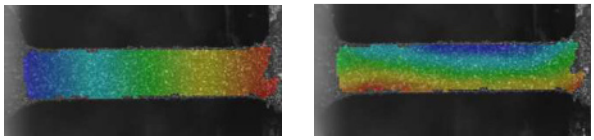
Fig. 3. Nano characterization system and DIC cameras.

The SU-8 processing consists of spin coat, soft bake, expose, post exposure bake (PEB), development and an optional hard bake. SU-8 was coated by static dispense followed by spread cycle for 10 seconds at 500 rpm and spin cycle for 35 ~ 40 seconds at 1350 ~ 2900 rpm. Time and speed during spin cycle varied depending on desired SU-8 film thickness. After spinning, the substrate was soft baked at 65°C for 30 minutes and then at 95°C for 90 minutes to evaporate the solvent and densify the film. After baking, the substrate was allowed to cool to room temperature and then exposed. UVP B-100 AP, 365 nm wavelength exposure tool was used for UV exposure. The intensity of mercury lamp was regularly checked for any inconsistencies. The mask, printed on a transparency, was placed on the wafer and then a glass plate was put over it. The wafer was exposed for 20 seconds in each dose step with 60 seconds interval between consecutive exposures. This allowed place, and avoided the hardening of the top surface that could better absorption of energy and light induced reactions to affect the film quality due to high exposure dose and long exposure time. After a hold time of about an hour, PEB was carried out for 2 minutes at 65°C and 20 minutes at 95°C to cross-link exposed regions. To minimize stress and resist cracking, temperature was increased in two steps. Then, the substrate was allowed to slowly come to room temperature and immersed in SU-8 developer to release SU-8 dog-bone samples from the substrate. An optional hard bake condition will be described in section 3.2 to investigate the effect of its temperature and time on viscoelastic material properties.

3. Experimental details

3.1 Tensile test

For the uni-axial tensile test, an in-house micro-mechanical tester, named nano-characterization system (NCS), and digital image correlation (DIC) technique [20] were used to measure Young's modulus and Poisson's ratio as a function of temperature and applied strain rate. Fig. 3 shows the components of NCS, which was designed and implemented with precision linear stages and a six-axis force transducer to accurately measure force for mechanical properties of thin film materials [21]. The DIC, a form of photogrammetry, is a non-contact optical deformation measurement technique in which the sur-



(a) X-directional displacement (b) Y-directional displacement

Fig. 4. Deformation contours from DIC cameras.

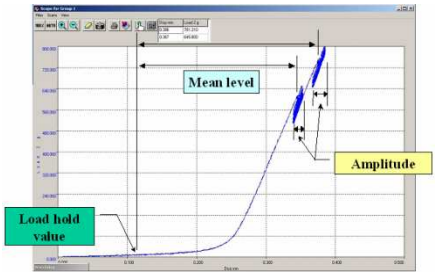


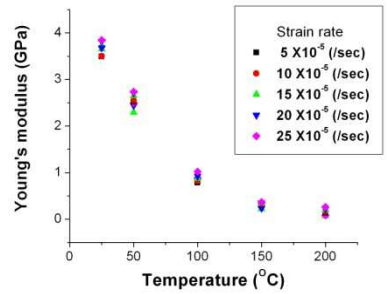
Fig. 5. Typical DMA testing profile.

face features of the object are traced in digital images. Even if there is a slippage between sample and grip at high temperature, DIC can measure accurate strain from local deformation. In addition, Poisson's ratio can be obtained through DIC technique since not only longitudinal strain but also transverse strain is captured simultaneously. It is noted that the specimen should be stretched prior to starting the test because the free-standing specimen buckles up while the environmental chamber is heated to target temperature.

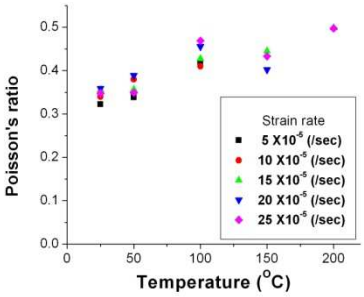
In this testing, applied strain rate is varied between 0.8 μ/sec and 5.6 μ/sec at elevated temperatures from 25°C to 200 °C. Six or seven DIC images were captured in the middle of testing to extract strain values at each stage, and at the same time the force read from load cell was recorded for stress value corresponding to strain value. In Fig. 4, the deformations in X- and Y-direction taken by DIC cameras are shown where red and blue color indicate the maximum and minimum, respectively. The increase of strain rate is necessary in order to get a clear relationship between stress and stain at high temperature, especially above the glass transition temperature.

3.2 DMA (dynamic mechanical analysis)

For DMA, Bose® ELF3200 [22] and WinTest DMA software [23] were used to measure storage/loss modulus and loss factor by applying an oscillating force to the sample. The load cell of 1000 gram was used. The parameters for DMA are temperature and frequency. The frequencies between 0.1 Hz and 10 Hz were considered in log scale at various temperatures from 25°C to 200°C. As mentioned, the specimen was stretched before starting DMA to remove the initial buckling that occurred during the preheating process. To ensure sample flatness, load hold value, which is the requested force used to establish the starting point for tensile test, was set to 10 gram. Fig. 5 shows a typical DMA testing profile at 150°C in a graph of displacement vs. force where load hold value, mean



(a) Young's modulus



(b) Poisson's ratio

Fig. 6. Elastic material property vs. temperature.

level and dynamic amplitude are designated. A mean level was chosen between 0.15 mm and 0.4 mm depending on temperature, and two kinds of dynamic amplitude of 0.015 mm and 0.02 mm were considered.

To investigate the effect of hard bake on viscoelastic material properties, the time condition was supplemented. The sample was kept in the chamber at a constant temperature between 75°C and 200°C for up to 4 hours, and DMA was carried out at every hour. Based on the DMA results, a hard bake condition was chosen to get stable material properties. After processing the hard bake, DMA was carried out once more by using the same testing conditions of temperature, frequency and time. Its results were compared with those without hard bake.

4. Experimental results

4.1 Elastic material properties

First, the variations of Young's modulus and Poisson's ratio according to temperature are shown in Fig. 6. As temperature increases, Young's modulus decreases and Poisson's ratio increases. From Fig. 6(a), glass transition temperature seems to be in the range of 100 ~ 150°C. It was confirmed from TMA (thermo-mechanical analysis) [24, 25] that glass transition temperature is in the range of 130 ~ 140°C. Fig. 6(a) is redrawn in Fig. 7 to show the effect of strain rate on Young's modulus. In all the temperatures, Young's modulus of SU-8 increases as strain rate increases. The effect of strain rate on Young's modulus is more remarkable at higher temperature. For Poisson's ratio, it is hard to find a tendency related to

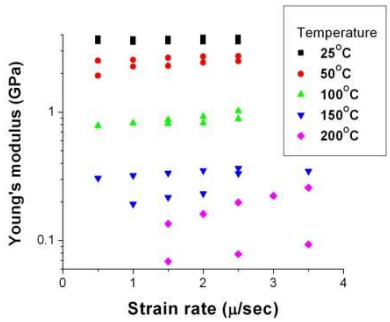


Fig. 7. Young's modulus vs. strain rate.

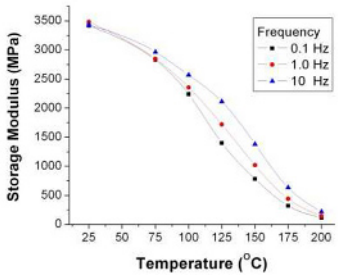
strain rate effect as shown in Fig. 6(b). That is, the effect of loading rate on Poisson's ratio can be negligible compared to that on Young's modulus.

4.2 Viscoelastic material properties

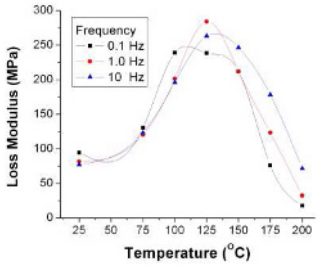
This section is composed of three parts: DMA of SU-8 sample without hard bake, DMA of SU-8 sample after additional thermal curing, and the DMA of SU-8 after hard bake is chosen from the results of the second DMA.

Fig. 8 shows storage modulus, loss modulus and loss factor (tan δ) according to temperature increase when dynamic amplitude is 0.02 mm. Generally, the abrupt fall of storage modulus and the peak of loss modulus (or tan δ) happen around the glass transition temperature [26]. Therefore, it can be double-checked that the glass transition temperature of SU-8 used in this study is between 125°C and 150°C. As frequency increases, the storage modulus increases and its increase is remarkable around the glass transition temperature. As the frequency increases, loss modulus above glass transition temperature increases and the peak of loss modulus (or tan δ) is shifted to the right. That is, the increase of frequency makes the glass transition temperature of SU-8 material increase.

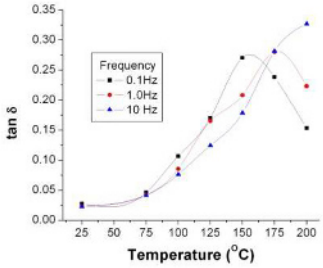
In Figs. 9-11, the variations of viscoelastic material properties are shown according to thermal curing condition at a frequency of 1 Hz. As temperature holding time (THT) increases, the storage modulus increases especially in the case of more than 100°C as shown in Fig. 9(a). Specifically, the increases of storage modulus after 4-hour THT above 125°C are around 700 ~ 900 MPa. SU-8 becomes stiffer as THT increases because SU-8 is cross-linked further due to additional thermal curing. Fig. 9(b) shows that storage modulus curve as a function of temperature is turned upward around 100°C as THT increases. Fig. 10 shows the loss modulus according to thermal curing. As THT increases, the loss modulus converges to a value at any temperature. In Fig. 10(b), the maximum of loss modulus is reduced and moved to the right as THT increases. From Fig. 11, loss factor decreases as THT increases. From the shift of maximum of loss modulus and loss factor, it can be said that glass transition temperature increases due to thermal curing. Since storage modulus, loss modulus and loss factor above 100°C seem to converge after 3 hours THT, 3-



(a) Storage modulus

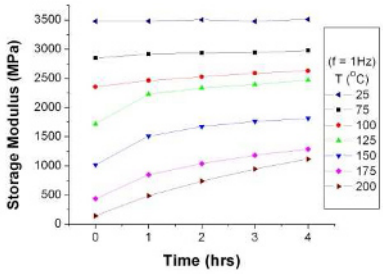


(b) Loss modulus

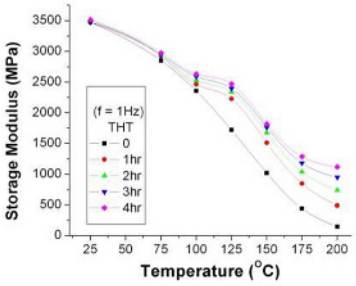


(c) tan δ

Fig. 8. Viscoelastic material properties of SU-8.

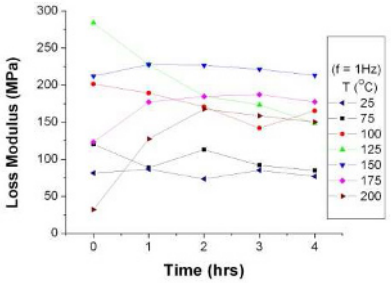


(a) Temperature holding time (THT) effect

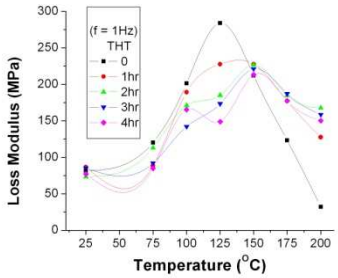


(b) Temperature effect

Fig. 9. Storage modulus according to thermal curing condition (f = 1 Hz).

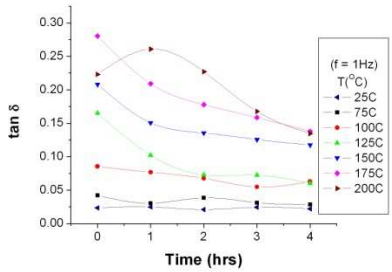


(a) Temperature holding time (THT) effect

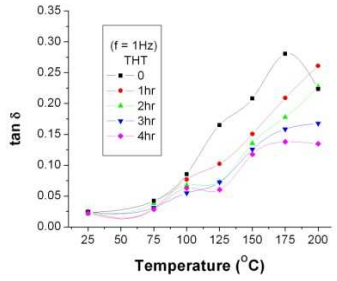


(b) Temperature effect

Fig. 10. Loss modulus according to thermal curing condition (f = 1 Hz).

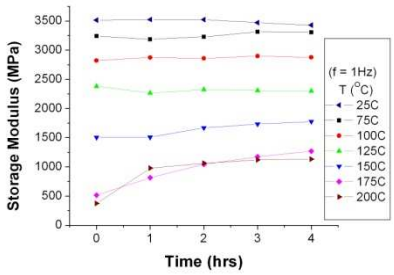


(a) Temperature holding time (THT) effect

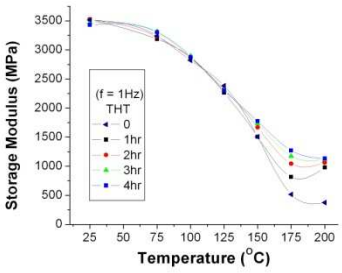


(b) Temperature effect

Fig. 11. Loss factor according to thermal curing condition (f = 1 Hz).

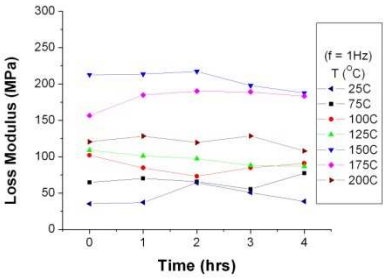


(a) Temperature holding time (THT) effect

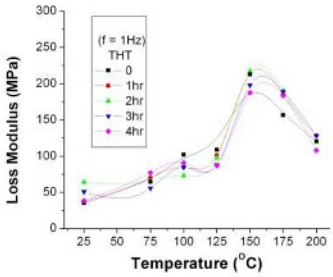


(b) Temperature effect

Fig. 12. Storage modulus after hard bake according to thermal curing condition (f = 1 Hz).



(a) Temperature holding time (THT) effect

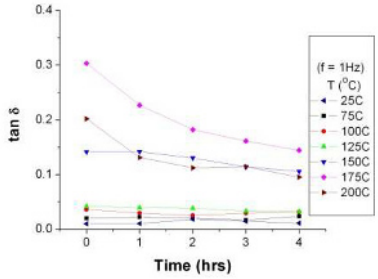


(b) Temperature effect

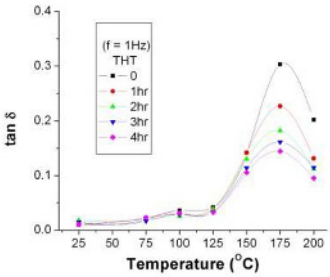
Fig. 13. Loss modulus after hard bake according to thermal curing condition (f = 1 Hz).

hours was chosen as a hard bake time. For a hard bake temperature, 150°C was used.

In Figs. 12-14, the viscoelastic material properties of SU-8 material after a hard bake of 150°C for three hours are shown according to thermal curing time and temperature. The storage modulus after hard bake shown in Fig. 12(a) is larger than the one without hard bake shown in Fig. 9(a). The dependency of storage modulus for THT is much reduced up to 150°C. That is, the storage modulus curve as a function of temperature does not change up to 150°C with regard to THT as shown in Fig. 12(b). Fig. 13 shows the decreased loss modulus compared to loss modulus without hard bake. The variation of loss modulus according to THT is also much reduced. From Fig. 14, the loss factor below 125°C is very small and its variation is also small. From the results after hard bake, it can be seen that hard bake process is necessary to stabilize viscoelastic



(a) Temperature holding time (THT) effect



(b) Temperature effect

Fig. 14. Loss factor after hard bake according to thermal curing condition (f = 1 Hz).

material properties of SU-8 polymer material to be used at high temperature above post exposure bake temperature.

5. Conclusions

The effects of temperature on mechanical properties of SU-8 were investigated to characterize SU-8 material in a broad range of temperature. Since SU-8 has many advantages for MEMS application and it can be used in a high temperature environment, the material characterization at elevated temperature is essential for the design of MEMS structures. The elastic and viscoelastic material properties were measured through tensile test and dynamic mechanical analysis. The effects of temperature and strain rate on Young's modulus and Poisson's ratio were observed. The effects of temperature and frequency on storage/loss modulus and loss factor were obtained. Since a hard bake could severely affect the material properties of SU-8 above post exposure bake temperature, hard bake conditions such as temperature and temperature holding time were varied to obtain optimal hard bake condition. After a temperature of 150°C was applied for three hours, the viscoelastic material properties below 150°C were maintained under additional thermal curing. Therefore, it is recommended that a hard bake process at maximum operating temperature is useful to guarantee consistent material properties at high operating temperature.

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