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Determining the physical properties of EpoClad negative photoresist for use in MEMS applications

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Abstract. In this paper, the internal stress and Young modulus of EpoClad are investigated using different methods. The swelling behavior when submerged in common processing liquids is also measured and reported. Different on-wafer MEM structures are used, together with nanoindentation measurements. The experimental results yield an internal stress that is strongly dependent on the processing conditions, the ambient and the submersion liquid. Young's modulus is found to be 4.4 +/- 0.2GPa.

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1. Introduction

EpoClad is a relatively new, negative tone photoresist produced by Micro Resist Technology GmbH [1]. Prior investigations [2] have shown that the mechanical properties of EpoClad resemble those of the more commonly used SU-8 [3], also an epoxy based resin. Over the last decade, SU-8 has emerged as a popular structural material for polymer micro electromechanical systems (MEMS), specifically in high aspect ratio applications such as microfluidic channels [4], pneumatic valves and actuators [5], optical waveguides [6] and mechanical springs [7]. In this study, EpoClad is characterized as a polymer that can be used for the fabrication of polymer MEMS. Polymers can decrease the production cost of MEMS and enable the exploitation of the versatile properties they have to offer, compared to the traditionally used silicon and metals [8]. These properties include high elasticity and large thermal expansion coefficients. With respect to other typical negative photoresists such as the aforementioned SU-8, EpoClad specifically offers superior adhesion properties to silicon substrates, a precisely tunable refractive index by combining it with EpoCore and excellent heat and pressure resistance [1].

EpoClad is a photodefinable epoxy. This means that the highly crosslinked polymer network of EpoClad provides a very stable plastic, both mechanically and chemically. However, in order to use this material as an active mechanical structure, a detailed study of its properties is mandatory. The internal stress of the EpoClad polymer is measured using on-wafer stress indicator structures and nanoindentation on chariot wheel structures [9]. As an expansion to previous work, the stress indicator structures are also used in swelling behavior tests. More specific, these are

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measurements of volume change of the polymer depending on the ambient. When the EpoClad is submerged in a liquid, this liquid can influence the polymer network. Any absorbed moisture will take up space in the EpoClad layer, thus inducing a compressive stress. The change in stress can then be recorded by the indicator structures. It is known that negative photoresists swell during development or in other circumstances and for SU-8 these effects were already investigated and modeled [10]. A purely quantitative study of this behavior in EpoClad will yield more insight in its properties and is mandatory when envisioning it as a permanent, structural layer in MEMS.

Since the degree of crosslinking in a polymer is a function of the processing parameters [11], the experiments to determine the internal stress are repeated using structures that underwent a slightly altered fabrication process.

Furthermore, the Young modulus of EpoClad is measured using standard continuous stiffness measurement (CSM) nanoindentations on thick EpoClad films and nanoindentations of chariot wheel structures. The results of these tests are compared with data from a standard macroscopic tensile test [2].

In the first section of this report, a short overview of the modeling of the on-wafer test structures and the fabrication is given. The second part describes, in detail, the testing of the indicator structures in different ambients, the nanoindentation experiments and the results.

2. Device modeling and manufacture

2.1. Stress indicator structures

The used stress indicator structures are an adaptation of the design proposed by Goosen et al. [12]. They produce a visually measurable displacement, depending on the stress present in the test beams before the structure is released from the substrate during the fabrication process. The working principle of these structures is illustrated and explained using figure 1. When the free-hanging test beams relax their built-in tensile stress, after the underlying sacrificial layer has been etched, they shrink. This very small difference in length is amplified by the amplification beams to produce a measurable displacement that can be accurately recorded using the Vernier grating. Devices with test beam lengths of 250 μm and 100 μm were used for the measurements, [13]. The average thickness of the beams used in this investigation was 19 μm .

When the structures are submerged in a liquid, the EpoClad polymer absorbs some of the submersion liquid and an additional stress component is induced that will either expand the test beams or shrink them even further. Hence, the stress in an EpoClad layer can be recorded as a function of the ambient.

Figure 1. A scanning electron microscope (SEM) image of a stress indicator structure with 100 μm test beams and a Vernier grating at the end of the amplification beams.

Through finite element simulations using the COMSOL Multiphysics Simulation Software, the relationship between measured displacement and internal stress was obtained. Models of the structures were created and a deviation-to-stress conversion factor was obtained. To simulate these models, some material characteristics had to be selected from available sources. Young's modulus E was chosen to be 2.2GPa, as determined by a standard strain test during prior research

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[2]. The internal stress was estimated at 10MPa and a value of 0.26 was used for the Poisson ratio as this is a typical value for polymers.

2.2. Chariot wheels

In this section, an analytical model for the chariot wheel structures is described. These structures were used in the derivation of the Young modulus and the internal stress of EpoClad. Some of the wheel structures are shown in figure 2. As indicated, the outer part of each chariot wheel is connected to the substrate. Each of the four spokes is suspended approximately $3\mu\text{m}$ above the surface. Spokes widths (W) are $40\mu\text{m}$ or $20\mu\text{m}$. Spoke lengths (L) vary between $900\mu\text{m}$ and $200\mu\text{m}$. The wheels were fabricated in two thicknesses (H): $17\mu\text{m}$ and $19\mu\text{m}$.

Figure 2. A SEM image showing four fabricated chariot wheel structures as part of a larger array with different spoke lengths. Structural thickness is $19\mu\text{m}$ and spoke width is $40\mu\text{m}$.

The analytical model is an adaptation of a more thorough discussion by Senturia [14]. For a double-clamped beam, such as each individual spoke, the stiffness can be written in terms of beam dimensions, the internal stress and the elastic modulus of the beam material. Table 1 shows the symbols used for these parameters.

Table 1. Parameter symbols

Parameter	Symbol
Applied force	F
Central displacement	c
Elastic modulus	E
Height (thickness)	H
Internal stress	σ_0
Length	L
Stiffness	k
Width	W

Deducting the analytical relationship is done by means of an energy optimization technique. The shape of the deformed beam is approximated by an arbitrary parameterized deformation function.

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The parameters in this function are determined so that the elastic energy of the beam and the work done for the deformation are minimal. As an example: when a vertical load is applied to the center (i.e. at $x = 0$) of a beam with length L , (1) is a suitable deformation function, $w(x)$. It automatically fulfills the boundary conditions of ideal mechanical clamping at $x = \pm L/2$.

$$w(x) = \frac{c}{2} \left[1 + \cos\left(\frac{2\pi x}{L}\right) \right] \quad (1)$$

Using this test function with c as the parameter to be optimized, via the basis for the energy method, will result in

$$F = \left[A \left(\frac{\sigma_0 WH}{L} \right) + B \left(\frac{EWH^3}{L^3} \right) \right] \cdot c \quad (2)$$

which relates the applied vertical force to the central displacement. All higher order terms of the center displacement were discarded. The theoretical values of the dimensionless parameters A and B in (2) are $\pi^2/2$ and $\pi^4/6$ respectively and are dependent on the choice of the initial deformation function (1).

With finite element simulations, different beam lengths were modeled. From the simulation data, the values of A and B were determined to be 3.55 and 16.1 respectively, which is only slightly different from the theoretical values mentioned above. The values resulting from the simulations were used for all further calculations.

Since the wheels are basically constructed out of four double-clamped beams, it is possible to use the stiffness of one such beam to define the stiffness of the entire wheel. An additional stiffness multiplier α is needed and (2) is changed to:

$$F = k_{wheel} \cdot c = \alpha \cdot k_{beam} \cdot c \quad (3)$$

with k_{wheel} and k_{beam} the stiffness coefficients of the chariot wheel and the individual beam respectively. Combining (2) and (3) finally results in:

$$k_{wheel} \cdot L = \alpha AHW\sigma_0 + \alpha \frac{BH^3W}{L^2} E \quad (4)$$

To determine the stiffness multiplier, finite element simulations of the chariot wheel structures with different spoke lengths were made. The stiffness of each wheel was simulated and compared to the stiffness of the corresponding single beam. For each length L , a value of the stiffness multiplier α was found. The evolution of α in function of L is shown in figure 3.

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Figure 3. Graph of the stiffness multiplier α in function of the spoke length L . For very short or very long beams, α converges to a fixed value.

The value of α converges to 4 for increasing beam length, the number of beams that make up a single chariot wheel. This convergence can be explained by the fact that the influence of the center of the wheel, where the beams join, decreases as the spoke length increases. For ever shorter beams, this center becomes more dominant. A more membrane-like behavior is observed and α also converges to a fixed value.

2.3. Fabrication

To start the fabrication of the structures, a silicon substrate was prepared by removing any organic residue in piranha etch (sulfuric acid and hydrogen peroxide). Although adhesion between the substrate and EpoClad was expected to be high enough, a layer of chromium was sputtered onto the dehydrated substrate to improve it even further.

A 3μm sacrificial polyimide layer (LOR 10B) was spin coated and patterned with a conventional photolithography step, using S1818 positive photoresist. The EpoClad was spin coated in two steps: a 10-second prespread at 1000rpm, followed by 60 seconds at 4000rpm. A thickness of approximately 19μm was obtained. The film was soft baked gradually using a conventional hotplate. After a 15-minute relaxation at room temperature, the hotplate was heated to and held at 50°C for 15 minutes. Following a third segment of 15 minutes at 90°C, the temperature was increased to 115°C and held for an hour. Finally, the hotplate was turned off and allowed to cool down back to room temperature slowly. Baking times were longer than recommended by the manufacturer datasheet in order to avoid the EpoClad film sticking to the mask during exposure. Gradual heating and cooling was used to avoid thermal shocks.

The 80-second UV illumination had an intensity of 5mW/cm² around the g-line of the spectrum (436nm). A post exposure bake was performed in a conventional oven under nitrogen atmosphere at 110°C. After baking for 60 minutes, the substrate was allowed to cool down back to room temperature gradually inside the oven for 90 minutes.

Patterning of the EpoClad layer was done with PGMEA (Propylene Glycol Methyl Ether Acetate), which was then rinsed with IPA (IsoPropyl Alcohol) and left to dry. To further cure the EpoClad, a one-hour hard bake was performed at 150°C, again under N₂ atmosphere. As for the post exposure bake, the substrate was allowed to cool down to room temperature inside the oven for about one and a half hour to avoid thermal shock.

The sacrificial layer was etched using an alkaline solution. Subsequently, the substrate was immersed in IPA before undergoing a CO₂ critical point drying (CPD) to obtain a stictionless release. During CPD, a residual thin film had formed on top of the structures. This residue was removed by means of a two-minute reactive ion etching (RIE) in oxygen plasma.

To study the influence of the degree of curing on the internal stress, a slightly different processing was used to fabricate an extra batch of stress indicator structures. First of all, the hard bake time was doubled to two hours. Since only a limited amount of residue had formed during CPD, on non-critical locations on the substrate, no RIE was performed on these structures.

3. Experimental details and results

3.1. Stress indicator structures

After fabrication, the dried indicator structures were inspected by means of an optical microscope. Photographs were taken and the deviation of the Vernier gratings was recorded. Measurement data was processed separately for structures with different dimensions. Results per set of indicators were then averaged and standard deviations were calculated.

In earlier research [13] the indicator structures were used to determine the coefficient of thermal expansion (CTE) of EpoClad. In this investigation however, the focus remains on the internal stress, both in dry and wet ambient. Therefore, the dry structures were also placed in a Petri dish and submerged in several liquids to determine the swelling behavior. The internal stress was recorded in PGMEA, IPA and water. The solvents play an important role in the fabrication process, as was mentioned earlier. Water has a very high influence on the internal stress and is always present in the form of humidity in the ambient air.

The first liquid, PGMEA, was poured onto the dry structures. To switch between liquids (e.g. from IPA to water), most of the first liquid was pipetted from the dish, but a minimal amount was left behind, to prevent stiction. The second liquid was then added in abundance and the structures were allowed some time to settle. After two to three minutes, the dynamic effects of swelling have vanished and the structures reach a steady state. Consequently, the deflection of the Vernier grating was recorded using an optical microscope and a digital camera. The transition itself was documented as well, but fluid mixing effects made it difficult to interpret most of the images.

3.2. Nanoindentation measurements on EpoClad

A standard continuous stiffness measurement (CSM) nanoindentation experiment was executed using a nanoindenter XP system from MTS Systems Corporation with a dynamic module and a CSM option under constant strain rate (0.05s^{-1}). A standard three-sided pyramid diamond indertip (Berkovich) was pushed into a flat layer of EpoClad [15]. While indenting, a small 75Hz oscillating signal was superimposed on the downward movement of the indertip. For indenting this EpoClad layer, CSM was preferred over a conventional nanoindentation. The conventional method focuses only on the unloading segment which, for a visco-elastic polymer such as EpoClad, could lead to an overestimation of the Young modulus. Continuously measuring stiffness in function of depth resolves this issue.

The CSM was executed on 30 different locations across the EpoClad layer. The Young modulus was calculated from the gathered data. For the calculations, a Poisson ratio of 0.26 was assumed. This is a typical value for polymers. Furthermore, it should be noted that because of the visco-elasticity of EpoClad, the stiffness of the material is a function of the indentation speed and the frequency of the superimposed signal. Briscoe [16] noted some other possible issues concerning the indentation of polymeric surfaces such as creep and relaxation [2]. However, these effects were negligible during our experiments.

The indentations performed on the chariot wheel structures were different from the ones performed on the EpoClad layer. Instead of a Berkovich geometry, a conical flat ended tip with a diameter of $10\mu\text{m}$ was used to apply a monotonically increasing load of 0.005mN/s to the center of the wheel. On every wheel, 15 consecutive measurements were performed to reduce variance. Applied loads and center displacements were automatically recorded. Afterwards, these were used to calculate the average stiffness per wheel. The resulting data were used to produce a graph representation of the kL versus $1/L^2$ relationship per set of wheels (same spoke width and height). Using a linear fit, shown in figure 4, and correlating this with (4), the Young modulus and the internal stress of EpoClad could be calculated.

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Figure 4. Linear fit through the indentation data of ten wheels with a height of 17 μm and a width of 40 μm . The line equation combined with (4) and values of the stiffness multiplier α (figure 3) results in values for internal stress and modulus of elasticity.

For the calculation of the stiffness of the beams in the chariot wheels, no estimation of the Poisson ratio was required. This is the main advantage of these seemingly complex structures compared to the simple EpoClad layer. Moreover, since the stiffness of a single beam would hardly be measureable, four beams were fitted into one chariot wheel structure. This not only increases the total stiffness, it also reduces the relative measurement error.

3.3. Results

Figure 5 presents the average measured values (and three standard deviations up and down) for the residual stress in dry EpoClad which was hard baked for one hour. Three sets of indicator structures were analyzed and three sets of chariot wheel structures were indented. Most of the results show an internal stress around 12MPa in the EpoClad, which is consistent with earlier published work in [13]. The thicker chariot wheels show a significantly lower internal stress, about 8MPa. Testing conditions, such as relative humidity of the air and temperature, may have a large influence on these results.

Figure 5. Results for the internal stress measurements. The indicators in set 1 were 17 μm thick with 250 μm test beams. Indicatorsets 2 and 3 were 19 μm thick with 250 μm and 100 μm test beams respectively. Wheelset 1 was 17 μm thick with spoke width 40 μm . Wheelsets 2 and 3 were 19 μm thick with spoke width 40 μm and 20 μm respectively.

Figure 6 shows the static results of the swelling tests on EpoClad stress indicator structures and reveals the influence of a longer hard bake (HB). The more thorough curing, caused by the additional thermal energy, generally leads to higher internal stress in all ambients. It should be noted that the reactive ion etching has an influence on the internal stress as well, albeit a limited

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one. Prior investigations on this topic revealed the etching process increased the stress present in epoxies [17]. The underlying physics however are still unknown.

When submerged in PGMEA or IPA, the internal stress increases, relative to the dry structures. Conversely, an additional compressive, internal stress caused by the absorbed water molecules, brings the total stress down to less than 25 percent of its original value in dry structures.

The solvents are thought to extract the moisture trapped inside the structures before the experiment, present in the ambient air, and thus increase the internal stress relative to the dry situation. When submerged in water, this liquid is absorbed by the polymer and causes it to swell. Or in other words, the absorbed water induces an extra compressive stress in the EpoClad, together with the built-in tensile stress. The net result is a stress in the film close to zero, relative to the dry situation. Figure 6 shows the average values calculated from the recorded indicator structure deflections after being submerged for at least three minutes, along with their standard deviations.

Figure 6. Comparison of the average internal stress in dry EpoClad indicator structures and structures submerged in various liquids. Results for structures processed with different hard bake (HB) times are shown. The prolonged curing introduces additional stress in the structures.

Using Vernier grating deflections recorded during the swelling of the polymer structures and applying an analytical model for the behavior, previously developed for the characterization of SU-8 [10], the dynamic swelling behavior of EpoClad was also charted. Figure 7 shows measurements of the test beam shrinkage during submersion of the dry structures in PGMEA. From the observations, poromechanical time constants τ_p were calculated. For the samples with a one-hour hard bake, a τ_p of 129 seconds was obtained. The more cured EpoClad was characterized by a τ_p equal to 150 seconds. Derivation of the initial bulk moduli yielded a K_0 around 98MPa for the less cured structures and 153MPa for the harder EpoClad.

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Figure 7. Visualization of the strain change (shrinking) of the test beams over time during the transition from dry to PGMEA. The dark circles represent samples with a one-hour hard bake. The light squares indicate data points for samples that were hard baked for two hours. Trend lines show that the more cured EpoClad shrinks more slowly.

Test beam expansion when the ambient is switched from IPA to water was more difficult to record due to mixing effects and thus less data points were gathered. However, the poromechanical time constants for expansion appear to be in the same range as those for shrinking, around 120 and 150 seconds. Initial bulk moduli are significantly higher (a factor of about 4) due to the denser structure of the dehydrated polymer.

In figure 8, the Young modulus shows very constant results for all executed test. Both the indentation of the plane via CSM and the conventional nanoindentation of different sets of chariot wheels return a value of 4.4GPa. All tested EpoClad structures were hard baked for 60 minutes. A prior macroscopic tensile strain test on EpoClad revealed a modulus of elasticity of 2.2GPa. This difference can be explained by several arguments. First of all, EpoClad is a visco-elastic polymer which makes it hard to determine one fixed elastic constant. The method of testing has a significant influence. The nanoindentation is a compressive test, or has a large compressive load component in the case of the chariot wheels. The strain test, however, applies a tensile load. Moreover, the built-in stress, which can change depending on the air humidity, can also affect the measurements of the Young modulus. For these reasons, it is not possible to state which testing method is the more accurate one.

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Figure 8. Results for the modulus of elasticity measurements. For the chariot wheels, the legend is the same as in figure 5. The EpoClad plane was 17 μ m thick. All samples were hard baked for one hour.

4. Conclusion

This article presented the detailed fabrication process, testing methods and a theoretical background to determine important material characteristics for micromachined polymers, applied to EpoClad negative tone photoresist, using on-wafer test structures. The internal stress was found to be approximately 12MPa in dry and room temperature conditions (RH 45% and 21°C). Internal tensile stress reached a maximum of 20MPa in PGMEA and similar values were recorded in IPA. Water absorption relieved the stress to just 2MPa. The swelling was characterized by a time constant of 129 seconds for layers of approximately 20 μ m thick.

The influence of a prolonged hard bake step to induce a more thoroughly cured EpoClad layer was investigated as well. The internal stress in dry structures increased to nearly 20MPa. In solvents, the stress build-up was more limited, but still rose to around 23MPa. Stress relief by immersion in water was not as effective as the total stress only dropped to approximately 5MPa. The poromechanical time constant characterizing the swelling for this harder EpoClad is approximately 150 seconds.

According to the authors' knowledge, this investigation is the first to quantitatively investigate the swelling behavior of EpoClad. It should be noted that the fabrication of the stress indicator structures is relatively simple. Thus, the swelling tests can be performed for other liquids when needed, or the influence of other process parameters can be verified.

The Young modulus was measured to be 4.4GPa using standard CSM nanoindentation and using indentation on chariot wheel structures made of EpoClad. The nature of these tests is mainly compressive or they have a significant compressive loading component and thus they offer a different insight than tensile strain tests into the mechanical properties of the visco-elastic EpoClad.

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