THERMO-MECHANICAL MATERIAL CHARACTERIZATION OF PHTOTORESISTS WITH HIGH ASPECT RATIO

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Summary: *MEMS made of photosensitive polymers offer new functionalities and applications in the micro world. Additionally to the new materials and advanced technologies the analysis of the thermo-mechanical material properties is required to ensure reliability and lifetime. Under this point of view, an uniaxial micro tensile measuring system has been developed to determine stress-strain-curves, Young's modulus or Poisson's ratio. The results will be shortly compared with those of Dynamic-Mechanical Analysis (DMA).*

Keywords: *Photosensitive polymers, material properties, microtensile test by DIC, DMA*

1 Introduction

Micro electromechanical systems (MEMS) based on silicon technologies are essential multipliers of technological progress during the last two decades. But also photosensitive polymers with high aspect ratio (HARMNST-materials) like SU-8 or Epocore are increasingly used for polymer MEMS like AFM cantilever arrays [1], components for micro air vehicles [2], polymer lenses [3] or force sensors [4]. To create new functionalities and applications the material behavior has to be characterized parallel to material development, process stabilization, design optimization or FE-simulation.

2 Measuring technique

Different measuring techniques among them atomic force microscopy or nano indentation have been established to analyze material parameters in micro-/nano-scale during the last two decades. But basic experimental techniques like uniaxial tensile test on micro specimens are under development until now. Under this point of view, a micro tensile measuring system has been developed and applied. It combines an air bearing based loading device, introduced by Sharpe and coworkers [5], and digital image correlation (DIC). Essential details of the measuring technique applied are described in [6, 7].

The geometry of the micro samples is adapted to the wafer technology. They are manufactured in batch processes on 4"- as well 6"-substrates. Fig. 1(a) shows the dimension of the dog-bone specimens. The thickness varies presently between $80\,\mu\mathrm{m}$ and $400\,\mu\mathrm{m}$ and depends on the resign mass dispensed on the wafer. The samples consist of epoxy-based negative photoresist Epocore made by micro resist technology GmbH Berlin or EPON® SU-8 from MicroChem Corp. Newton.

To achieve high contrast speckle patterns on the transparent specimen surface a thin reflecting texture is sprayed by airbrush, Fig. 1(b). This gray code distribution is recorded load controlled by DIC-system ARAMIS 2d from GOMmbH Braunschweig. The speckle pattern is captured via microscope. The highlighted area in Fig. 1(c) ensures the calculation of the in-plane displacement and strain fields, here in load steps of $0.1\,\mathrm{N}$.

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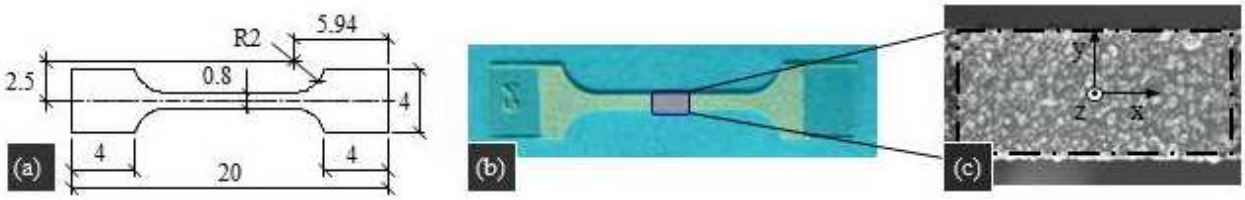

Figure 1: Micro sample: (a) geometry, (b) speckle pattern on the transparent surface with rectangle marked area for DIC measurement. (c) with shaded area for correlation analysis

Furthermore, the Dynamic-Mechanical Analysis (DMA) allows the analysis of thermo-mechanical material properties like storage modulus, loss tangent or glass transition region. These parameters can be determined as function of time, temperature or frequency. Therefore, the thin strip specimens with a length of $24\,\mathrm{mm}$, a width of $4\,\mathrm{mm}$ and a variable thickness pointed out at the micro samples for tensile test are sinusoidal loaded in tension mode. The experiments have been carried out at a temperature range between $-50\,^{\circ}\mathrm{C}$ and $200\,^{\circ}\mathrm{C}$ coupled with a heating rate of $1\,\mathrm{K/min}$ and a frequency sweep using the Dynamic-Mechanical Analyzer DMA 242C from NETZSCH Gerätebau GmbH Selb.

3 Results

In addition to the optical interferometric strain/displacement gage (ISDG) used by Sharpe [8] the DIC-technique allows to determine both the global and local deformation behavior on the surface of the micro samples. Fig. 2 shows selected strain fields ϵ_x(x, y) and ϵ_y(x, y) of a micro tensile specimen during loading. These fields are not completely uniformly distributed. They are more or less inhomogeneous. Basic strain domains occur. But, they are preferably located in the same surface region [6, 7]. To compress information

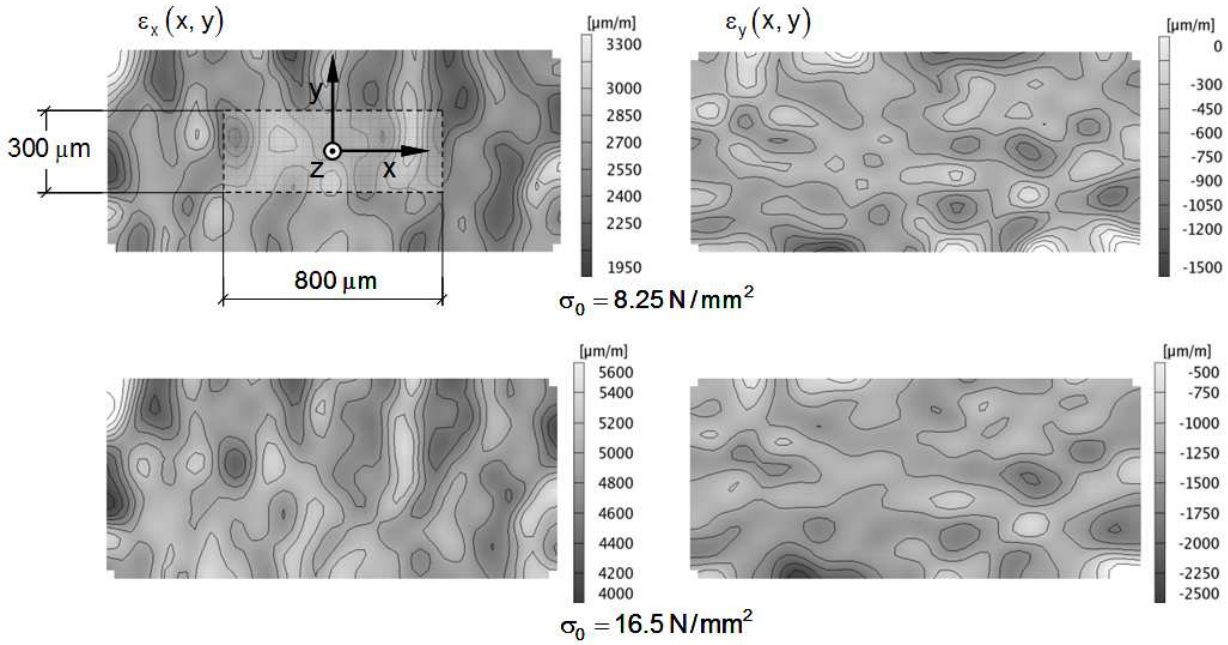

Figure 2: Strain fields ϵ_x(x, y) and ϵ_y(x, y) measured at two selected nominal stresses stages of sample s6-9. The evaluation area $\mathrm{A^{DIC}}$ for material parameters analysis is marked by the dashed rectangle.

and to obtain required material properties like stress-strain-curves, YOUNG's modulus or POISSON's ratio

a correlation adapted evaluation area A^{DIC} has been introduced, which is comparable with the mode of operation, known from the electrical strain gage, Fig. 2. Its area can be matched to the dimension of the micro sample. This approach enables to merge a certain number of measuring points, here approx. 300 points, and to calculate the mean value and the standard deviation for each load step.

The curves of the strain components in longitudinal and lateral direction are plotted against the applied load of sample b3-4, which consists of pure resist, Fig 3. They are sufficiently straight. The standard deviation of nearly $\pm 100\,\mu\mathrm{m/m}$ is small. Larger deviations up to $\pm 700\,\mu\mathrm{m/m}$ have been observed for larger strains in the case of a photoresist which is filled with pigments to modify the optical properties of the material. The stress-strain-diagrams of similar processed micro samples made of pure photosensitive polymers are comparable and comparatively linear up to a strain of $5 \cdot 10^3\,\mu\mathrm{m/m}$, Fig. 4. A clearly changed diagram has been obtained for the filled material. This filler acts as softening agent and leads to a reduced slope of stress-strain-curve. Due to the reduced thickness, the complete σ_x-ϵ_x-curve could be determined up to failure at nearly $47.5\,\mathrm{N/mm^2}$.

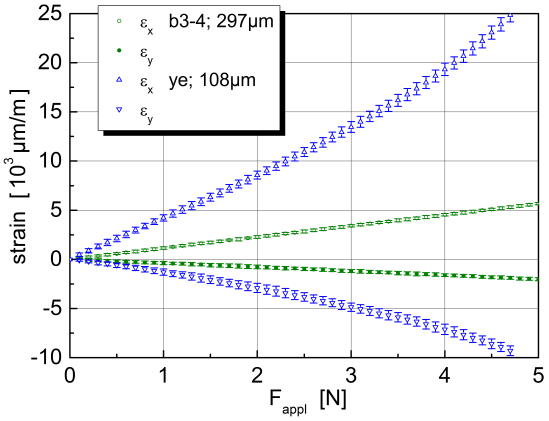

Figure 3: Strain components ϵ_x(x, y), ϵ_y(x, y) and their standard deviation as function of load

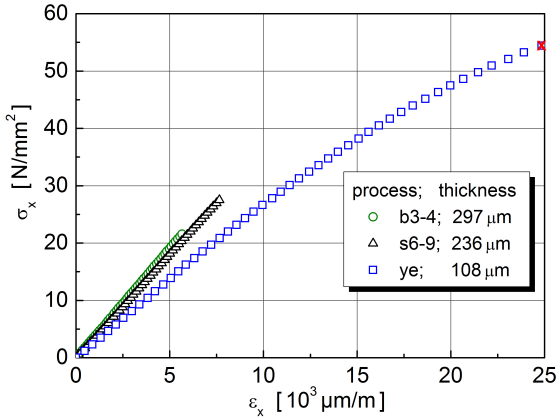

Figure 4: Stress-strain-diagrams of different lots

These results will be also found in the material parameters YOUNG's modulus E^{DIC} or POISSON's ratio ν^{DIC}, Fig. 5. E^{DIC} of lots b3-4 and s6-9 are in the range of $3.6\,\mathrm{GPa}$ with a variation of approx. $\pm 0.1\,\mathrm{GPa}$. It reaches a sufficient constant value from $2 \cdot 10^3\,\mu\mathrm{m/m}$. In contrast, the YOUNG's modulus is clearly reduced in the case of the filled photoresist. Similar effects can be observed for the POISSON's ratio whose determination is generally a sophisticated challenge. The measured strain dependent values scatter appreciable more in comparison to E^{DIC}, Fig. 6. This effect is reduced for longitudinal strains larger than $2 \cdot 10^3\,\mu\mathrm{m/m}$ for non-modified resists. But in the case of filled photosensitve polymer a constant could not be proved. The reason may be that weak material can be transferred between the molecular chains of the amorphous polymer. Considering the present experience these material parameters should be determined for longitudinal strains larger $0.1\,\%$ or better $0.2\,\%$ ($2 \cdot 10^3 \mu m/m$). An analysis window between $2...4 \cdot 10^3 \mu/m$ leads to practicable results, shaded window in Fig. 6. Consequently, the standards applied in the macroscopic world should be modified as technical compromise in the microscopic one.

Additionally, thermo-mechanical results have been obtained by the dynamic mechanical analysis. The storage modulus E' of SU-8 100 specimen decreases continuously as function of temperature starting from $3.8\,\mathrm{GPa}$ at $-50\,^\circ\mathrm{C}$ up to $200\,\mathrm{MPa}$ at $200\,^\circ\mathrm{C}$, Fig. 7. A constant level of E' could not been observed. To define reliable operating conditions and to estimate limits for functionality the knowledge of temperature T_g of glass transition region is required. The onset on storage modulus and the peak in loss tangent $\tan\delta$ describe this region. Furthermore, E' of b3-4 shows a distinct frequency dependence, Fig. 8. At $50\,^\circ\mathrm{C}$ it

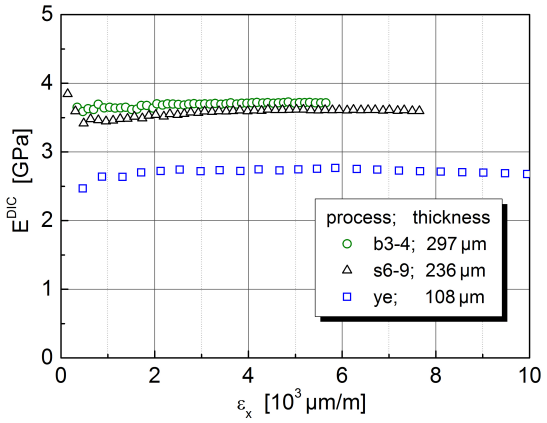

Figure 5: Strain dependent YOUNG's modulus

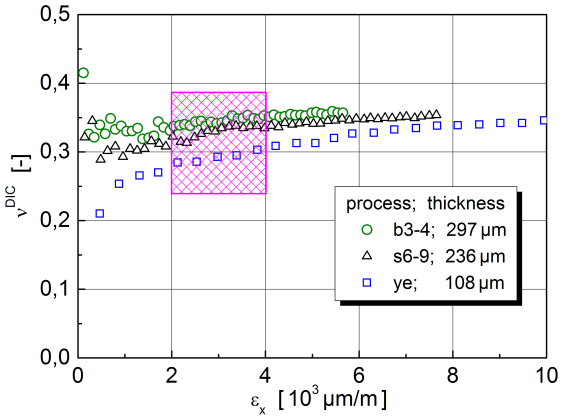

Figure 6: POISSON's ratio as function of process

starts to decrease rapidly. The small increase, starting at nearly $110\,^{\circ}\mathrm{C}$, may be caused by a post curing due to applying the temperature profile of the DMA-furnace. Thus, a thermal post treatment (tpt) results in a clearly modified, but more suitable storage modulus for different applications. Both, different materials SU-8

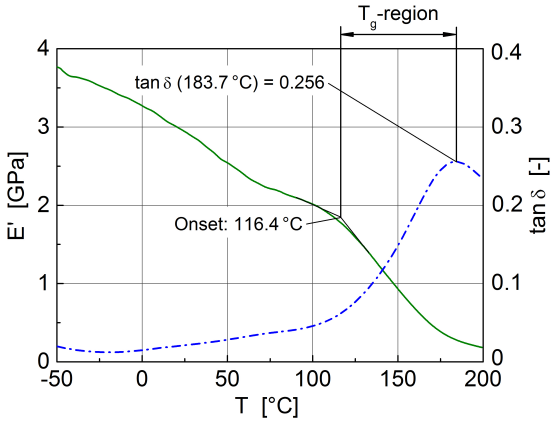

Figure 7: Storage modulus E', loss tangent $\tan\delta$ and region of glass transition temperature T_g of SU-8 100 sample c11 s3-4 tpt at 1 Hz

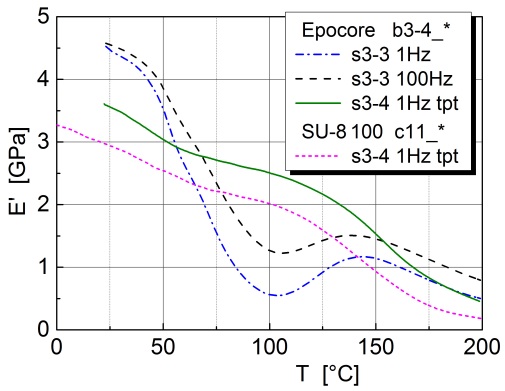

Figure 8: E' as function of frequency, thermal post treatment and different photoresists

and Epocore as well as variations in the technological process parameters lead to clearly visible differences in the temperature dependent storage modulus, Fig. 8. The root cause for this material behavior has to be analyzed.

4 Acknowledgment

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