# Experimental Analysis of Thermo-mechanical Material Properties of Photosensitive Polymers

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***Abstract:*** Micro tensile test and Dynamic Mechanical Analysis (DMA) are used to evaluate the material behaviour and several process steps of photosensitive polymers. DMA allows the determination of storage modulus, loss tangent or glass transition region as function of time, temperature and frequency. Furthermore, a sensitive loading device has been developed to carry out tensile tests at polymer micro samples. Stress-strain-curves, YOUNG's modulus or POISSON's ratio are analysed by Digital Image Correlation. The results are suitable to modify the process parameters, to characterise thermal and mechanical properties and to develop new application fields.

***Key words:*** *polymer MEMS, photoresist technology, dynamic mechanical analysis, uniaxial tensile test, digital image correlation, material parameter.*

## 1. Introduction

Silicon based Micro Electro Mechanical Systems (MEMS) as sensors, actuators or communicators became essential multiplicators of technological progress during the last two decades [1-7]. They integrate chemical, physical, biological and/or mechanical functionalities in one specialised system at milli- or micrometre scale. Driven by the demand of innovative applications, sustainability and energy efficiency alternative technologies are developed at the end of the last millennium to extend the application of photosensitive polymers in the field of functional MEMS.

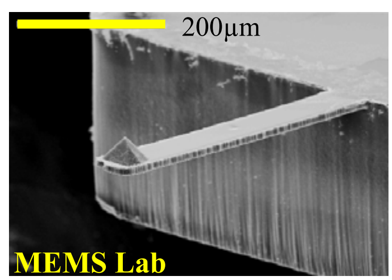


Fig. 1. *SEM-picture of an AFM-cantilever made of SU-8*

Such photoresists like SU-8 are used as mask for etching or as negative form for electroplating of metals to manufacture 3d-structures on wafer level. In addition, photoresist allows to process complex structures with high aspect ratio [8-12],

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like hands of watches, chip carriers for pyro-electric sensor chips or cantilever for atomic force microscopes (AFM), Fig. 1.

To generate new f unctionalities and application fields for polymer MEMS the knowledge of their thermo-mechanical properties is required in p arallel to th e design, material development and optimisation of the technological processes [10]. In the follo wing, two methods, Dynamic Mechanical Analysis (DMA) and micro tensile test, are selected to characterise the material behaviour of photosensitive polymers considering available recourses.

## 2. Process

The process run is s hown in Fig. 2. Two fabrication steps are essential: the re producible layer application and the drying process itself. To achieve layer of uniform thickness the resist is coated by a two axis robot and a dispenser on a 4 inch silicon wafer.

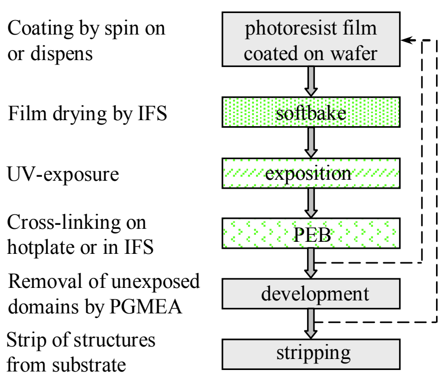


Fig. 2. *Flow chart of the process run for structure made of p hotoresists. Additional steps for multilayer structures marked by dashed lines.*

To get reproducible and homogeneous polymer structures an Infrared Furnace System (IFS) has been developed [10]. The IFS is a radiation based thermal equipment up to 150 mm substrate diameter. It affords to check the residual solvent during resist drying by gravimetric measurement in definite time intervals. The minimal resist mass is 500 mg and its resolution limit nearby is 10 mg. The IR-radiators are adjusted to the absorption bands of the resists in a wide wavelength region between 2 µm and 50 µm.

The system has the advantage to avoid the use of a hotplate, to be independent from substrate and resist type as well as to consider the initial mass including the available solvent. The reproducibility of the solvent loss during drying is the background for an improved handling of multilayer systems for 3d-structures. Different process steps like softbake, exposition or post exposure bake (PEB) have been tested to analyse their influence on the structures and the related material behaviour.

## 3. Micro tensile test

Different tensile test d evices are applied to analyse material properties of micro samples during the last decade [12-15]. Following a proposal of LaVan [13], an air bearing based device for mi cro specimens has been developed and used, Fig. 3.

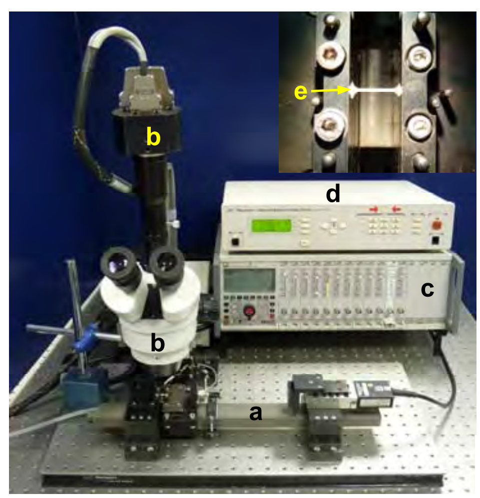


Fig. 3. *Micro tensile test system; a) air bearing with sample, load cell and translation stage, b) microscope with CCD-camera, c) MGC, d) NEWPORT universal motion controller, e) shouldered bar*

### 3.1. Experimental setup

The air bearing is the central tool of the loading device. Around the stator a high precision translation stage from Newport Corporation Irvine is arranged on the right sight. By this component the sliding table is moved via a thin cable. The applied force is measured by means of a 20 N load cell from Newport Electronics GmbH Deckenpfronn. The shouldered bar is located between the fixed and movable side by clamps on the same geometric plane.

Fig. 4 shows the bar geometry. The large paddles are required to have a sufficient contact area. The samples are made of photoresist EpoCore from mrt GmbH Berlin. To obtain a high-contrast textured pattern on the reflective surface of the transparent specimen an extremely thin granular pattern is coated by airbrush.

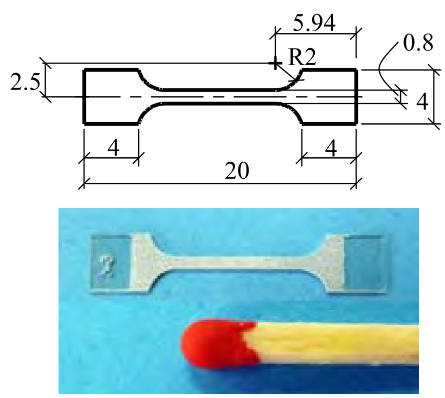


Fig. 4. *Geometry of shouldered test bar and manufactured sample with grey code*

This load device is combined with a Digital Image Correlation (DIC), here ARAMIS from GOMmbH Braunschweig, to measure the deformation on the sample surface [16-18]. To achieve the required magnification the CCD-camera is arranged on a stereomicroscope. The alignment of the clamped sample is a sophisticated process. The captures are taken load controlled using the output voltage from the load cell as trigger signal for ARAMIS.

To eliminate instabilities the sample is carefully preloaded with 0.2 N in load direction, x-axis in Fig.5. The very small kinetic friction in the air bearing is approximately neglected. Because the deformation expected is extremely small, the setup is placed on vibration isolated table.

### 3.2. Results

To obtain displacement and strain fields the capture of each load step is compared with the unloaded reference capture. The coordinate system is located in the middle of the sample, Fig. 5. The displacement component $u_x(x,y)$ at a load of 1.5 N is nearly uniform. But the appropriate strain fields $\varepsilon_x(x,y)$ and $\varepsilon_y(x,y)$ are not completely homogeneous. Strain domains occur. One idea to explain this effect may be that the resist and the hardening are not homogeneously distributed in the volume.

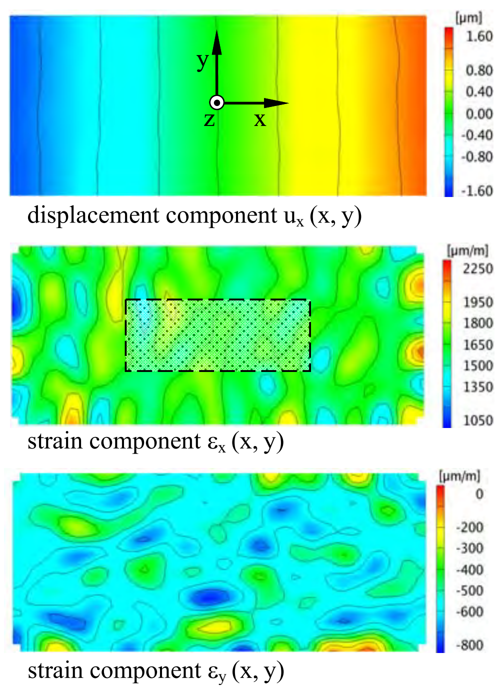


Fig. 5. *Selected displacement and strain fields at nominal stress* $\sigma_0 = 6{,}01\,\mathrm{N/mm^2}$ *of shouldered bar bb_2-4_s5-3*

To reduce the influence of the strain domains and to consider the statistic distribution of the grey code, the hatched area is analysed. The strains used in the following are the mean values of approx. 300 points comparable with the area of a strain gauge.

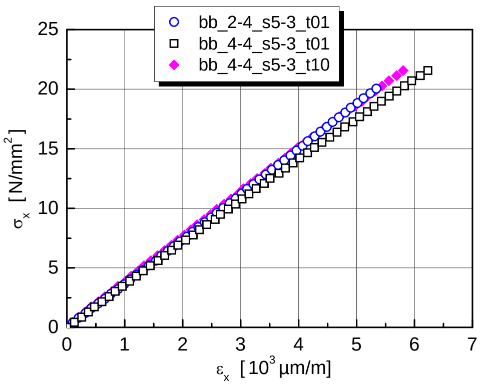


Fig. 6. *Stress-strain-curves of two lots*

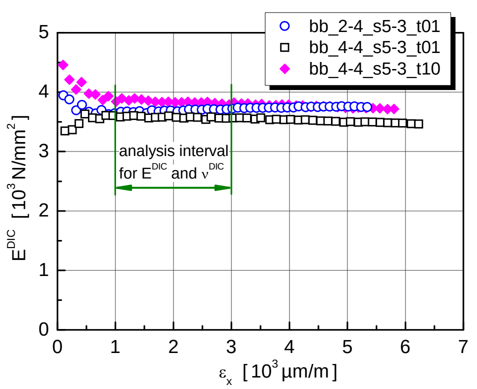


Fig. 7. *YOUNG's modulus by DIC*

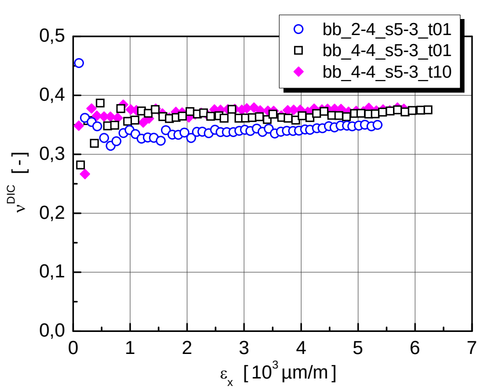


Fig. 8. *POISSON's ratio*

The stress-strain-curves of two different lots bb_2-4_* and bb_4-4_* as well as different test numbers vary not essentially, Fig. 6. Caused by a multiple loading the molecular chains are stretched and the material becomes stiffer. The YOUNG's modulus $E^{DIC}$ and POISSON's ratio $\nu^{DIC}$ are calculated between 0.1 % and 0.3 % strain following ASTM D3039 / D3039-M, Fig. 7 and 8. Modifications of the process parameter generate verifiable differences in the material parameters.

## 4. Dynamic Mechanical Analysis

Polymers have distinct temperature dependent properties. Characteristic material parameters are storage modulus E', loss modulus E", loss tangent $\tan\delta$ or region of glass transition temperature. Dynamic Mechanical Analysis (DMA) is a suitable method to determine them as function of time, temperature or frequency [18].

### 4.1. Experimental equipment

The system DMA 242C from NETZSCH Gerätebau GmbH Selb allows the parameter analysis using a set of load devices among them three point bending, compression or shear mode. The tension mode enables the measurement at thin strip sample processed on a wafer, Fig. 9.

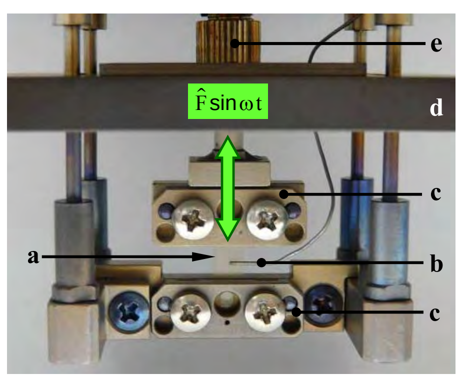


Fig. 9. *Tension device of DMA 242C a) sample, b) thermocouple, c) clamp, d) radiation shield, e) push rod*

The strip dimension is 24 mm x 4 mm x h. The thickness h varies between 150 µm and 350 µm depending on the mass of photoresist dispensed. The thickness of a sample can vary around some microns. The length between the clamps is 5 mm. The screws are tightened with a torque moment of 40 cNm. The sample is loaded by a sinusoidal force. The mean load of 1 N and the load amplitude of 0.5 N ensure a fluctuating tensile stress cycle. The heating rate ΔT is usually 1 K/min. In the case of frequencies smaller than 1 Hz ΔT is reduced to 0.1 K/min.

### 4.2. Results

The storage modulus E' is mainly reproducible in the whole temperature range, Fig. 10. The difference of E' between -150 and 100 °C is large with approx. 4500 N/mm$^2$. But, the curves decrease only by 1000 N/mm$^2$ between -50 °C and 50 °C.

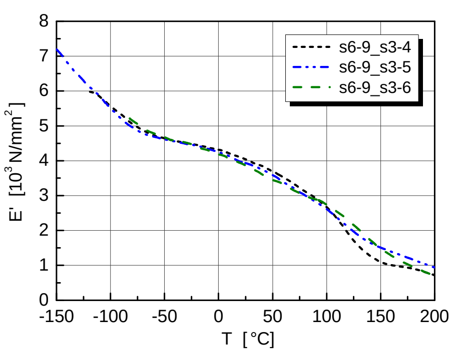


Fig. 10. *Storage modulus E' of three specimens from one wafer*

The storage modulus is frequency dependent over three decades, Fig. 11. E' differs less than 150 N/mm$^2$ in the temperature range from -50 °C to 50 °C. The photoresist becomes stiffer with increasing temperature especially for T > 50 °C. This effect is caused by the molecular chains in the polymer which are not able to follow the cyclic loading.

Furthermore, changes of process parameters like time for softbake, exposure or PEB during manufacture can lead to modification in storage modulus, Fig. 12. E' of sample bb_2-4_s3-3 is similar to s6-9_s3-5 up to 50 °C. Then, E' decreases below 500 N/mm$^2$ at 110 °C. A recovery occurs due to a further crosslink of the molecular chains. Therefore, storage modulus increases more than twice at 160 °C. To get an idea on the influence of thermal post-treatment sample bb_2-4_s3-2 is annealed using the temperature profile of Fig. 12. Then, the E'-curve is similar to lot s6-9. Simultaneously, this curve is comparable to that in photoelasticity applied Araldite B up to 100 °C.

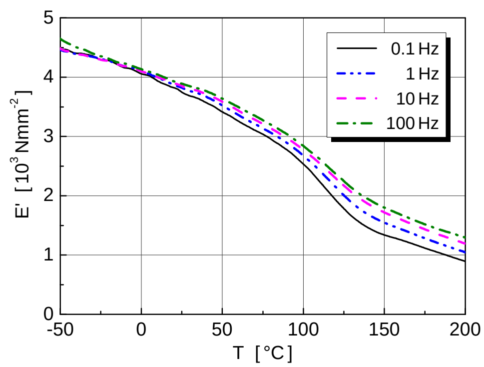


Fig. 11. *Storage modulus E' as function of frequencies used*

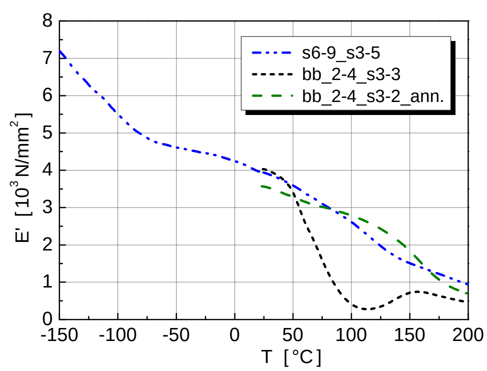


Fig. 12. *Storage modulus E' due to different process conditions*

### 5. Conclusions

Functional photoresists possess interesting thermo-mechanical material properties which are clearly different to silicon. The down scaling on wafer level requires also adapted analysing methods. A powerful measuring system combining micro uniaxial tensile test and DIC has been developed. This setup allows the noncontact analysis of essential material parameters like YOUNG's modulus or POISSON's ratio. The dynamic mechanical analysis shows that the material behaviour is distinctive temperature and frequency dependent. Modifications in the manufacturing process lead to more or less essential changes in the material properties.

## Acknowledgements

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