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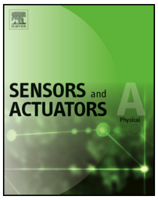

Optically and rheologically tailored polymers for applications in integrated optics

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ABSTRACT

We demonstrate an epoxy acrylate based system on which various micro-optical applications can be realized. With this innovative and flexible material concept, it is possible to adjust viscosity ($3\,\text{mPa s} < \eta < 46\,\text{Pa s}$) as required for different microstructuring processes e.g. inkjet printing, spin coating, and stereolithography and simultaneously precisely tailor refractive indices ($1.518 < n_{D,20} < 1.569$) which is essential to implement optical sensor concepts. The practicability of this material system is demonstrated for optical waveguide production and selected applications. The fast and low cost modification of optical and rheological material properties is advantageous for the fabrication of micro-optical polymer devices including planar-optical polymer sensor networks.

1. Introduction

For polymer based optronic systems, suitable polymer materials with defined physical and chemical properties are strongly needed which then path the way for applications in sensing, signal processing and data transmission [1–7]. Starting in the early 1960s, functional polymers lead to remarkable success in this field, leading to the development of a variety of different properties for applications in photovoltaic devices, LEDs, organic thin films or biomedicine [2,8–10]. They are now widely used in functional films, telecommunication applications, micro-optical components, and optical sensor concepts [6,11–15].

Refractive index is one of the key material parameters which has to be controlled and adjusted in such materials with regard to the realization of optical sensor systems, light sources and detectors or the substrate on which the required waveguides are deposited. Different approaches have so far been shown to modify the refractive index, such as the partially complex inorganic-organic multi-component hybrid materials for optical applications, as reviewed by Carlos et al. [5]. Another approach is the expansion of the polymer matrix with ceramic nano-particles like titanium dioxide. This is, however, difficult because these particles can agglomerate which then leads to an increase in optical damping due to scattering [16–19]. Avoiding huge effort and complex synthesis, the adequate choice of a co-monomer as well as the addition of guest molecules like phenanthrene are a simple and low cost method for refractive index adjustment [20].

With regard to the different production techniques of polymer devices (e.g. waveguide structures, feature sizes down to 10 μm), the viscosity of the material systems from which they are fabricated is a critical factor. Inkjet or offset printing, nanoimprint lithography, and reaction molding are "cold" processing methods where the monomer is shaped before UV-light or thermal induced polymerization is started. These processes have seen an increase in importance for optical waveguide fabrication [7,21,22]. While inkjet printing requires viscosities of around 50–500 mPa s [23], a viscosity around 200 mPa s suffices for offset or flexo printing.

In this work, the influence of the comonomer benzyl methacrylate in combination with Syntholux, a commercially available epoxy methacrylate, on the rheological, optical and thermomechanical properties of the compound material is investigated. This study is based on earlier investigations on the epoxy acrylate system [24],

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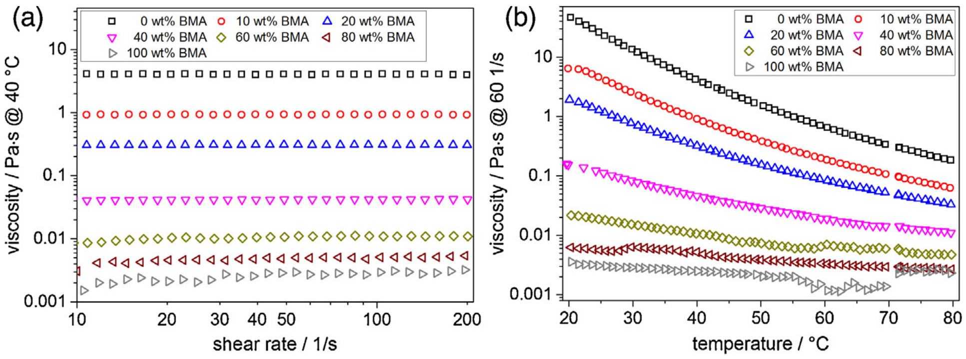

Fig. 1. Viscosity of mixtures of Syntholux and BMA measured with a cone and plate rheometer: (a) shear rate dependent and (b) temperature dependent.

enabling a wider range of applications of this material system in future.

2. Material system and characterization method

2.1. Materials and sample preparation

Three different monomers were used as starting material of the material system developed here. Syntholux, a commercial epoxy methacrylate, was used as main matrix monomer. Viscosity adjustment was done by using a suitable co-monomer. For UV curing a photo initiator was added. All used materials and their function are shown in Table 1.

The materials were mixed with a high speed stirrer (Ultra-Turrax T10, IKA, Staufen, Germany) under ambient conditions until a homogenous mixture was obtained. Polymerized samples made out of the different mixtures were produced for refractive index, optical damping and glass transition temperature measurements. For this, a setup made from glass plates, a silicone casting mold and FEP (fluorinated ethylene propylene)-foils was used. Polymerization was performed using a wavelength of 405 nm.

2.2. Characterization

The viscosity of the material system was determined by two distinct experiments with a cone and plate rheometer (CV50, Bohlin, Herrenberg, Germany). The first measurements were taken at a constant shear rate of 100 s^{-1} over a temperature range between 20 and 80 °C. Following this, the shear rate was swept logarithmically from 10 s^{-1} to 200 s^{-1} at temperatures of 20 °C, 60 °C and 80 °C. For quantification of refractive indices, an Abbe refractometer (DR-M2/1550, Atago, Tokyo, Japan) set at a temperature of 20 °C was used at three different wavelengths (450, 589, and 680 nm). Abbe numbers were then calculated. Optical damping values of the polymerized samples and materials, respectively, were obtained by gathering data with a UV–vis spectrophotometer (Cary 50 UV–vis, Varian, Waldbronn, Germany). This data was normalized by the sample thickness and corrected by the reflection losses occurring at the two interfaces of the sample and the surrounding air. The glass transition temperature (T_g) of polymerized samples was measured under nitrogen atmosphere using differential scanning calorimetry (DSC) (Phoenix 204F1, Netzsch, Selb, Germany). The temperature was increased from -80 °C to 200 °C with a heating rate of 10 K/min.

3. Results and discussion

3.1. Material fabrication

3.1.1. Flow behavior

The evaluation of the viscosity measurements shows a Newtonian behavior (shear rate independent) for all mixtures, as seen in Fig 1 (a) and for all investigated temperatures. On the other hand, the viscosity was found to be strongly influenced by temperature and BMA content as it is shown in Fig. 1 (b). At 20 °C, pure Syntholux shows a high viscosity of 46 Pa s which decreases to 0.18 Pa s at 80 °C. Pure BMA, conversely, shows a fairly low viscosity of 3.5 mPa s at 20 °C, which is clearly measured at the limit of the rheometer sensitivity range. BMA content dependence of the system can be also seen with a viscosity of 26 Pa s at 25 °C for pure Syntholux which decreases down to 3.1 mPa s for pure BMA. This data clearly shows that the material system can be adjusted to the various needs of the different microstructuring methods like inkjet or offset printing.

3.1.2. Optical and thermomechanical properties

The refractive index of the analyzed material system increased linearly with increasing BMA content, starting at $n_{D,20} = 1.550$ for pure Syntholux and reaching $n_{D,20} = 1.569$ for pure BMA (Fig. 2 (a)). Based on this data, the refractive index can be adjusted with high accuracy. Deviations in measured values are a result of sample fabrication and measurement principle. Air bubbles or dust in the sample or between sample and main prism of the Abbe refractometer can compromise measurement results [25]. Abbe numbers which were then calculated from the refractive index data are around 35 and are not influenced by BMA (Fig. 2 (b)). Optical damping of the polymerized samples strongly correlates with BMA. Samples out of pure Syntholux show a damping of 3 dB/cm at 600 nm which decreases to 0.5 db/cm for samples containing 80 wt% BMA (Fig. 2 (c)).

Merging the viscosity and refractive index data of the presented material system, a clear dependence of refractive index on viscosity can be seen and is shown in Fig 3. The same is done for the data of the material system presented earlier consisting of Syntholux and EGDMA [24]. With this co-monomer, the refractive index, in contrast to the case with BMA, linearly decreases with increasing co-monomer content. A total change in refractive index of up to 0.05 is possible, reaching a maximum of $n_{max} = 1.568$ and a minimum of $n_{min} = 1.518$ with the respective co-monomer, while still maintaining the same viscosity. With these combined material systems, for processes where viscosity is critical, it is now to realize optical waveguides where core and cladding require different refractive indices while viscosity must be constant.

For the potential applications of the material system, for example in optical sensor networks, the continuous operating temperature is of great importance. This temperature is somewhat lower than the glass transition temperature, and for a short period of time and without external stress the material can also be used at slightly higher temperatures. There are three points, which can be calculated for the glass transition temperature within the Proteus Thermal Analysis Software (Netzsch, Germany). The first and second point are denoted as the midpoint and the onset, respectively, and are determined by the tangent method, whereas the third is the inflection point [26,27]. When looking at the inflection point,

Table 1
Used materials, their abbreviations and functions as well as the respective suppliers.

Material	Abbr.	Function	Supplier
Syntholux		Main matrix monomer	Synthopol Chemie, Buxtehude, Germany
Benzyl methacrylate	BMA	Co-monomer	Sigma-Aldrich, Taufkirchen, Germany
Ethylene glycol dimethacrylate	EGDMA	Co-monomer	Sigma-Aldrich, Taufkirchen, Germany
Diphenyl(2,4,6-trimethylbenzoyl) phosphine oxide	DPO	Photo initiator	TCI chemicals, Eschborn, Germany
Irgacure 184		Photo initiator	BASF, Ludwigshafen, Germany

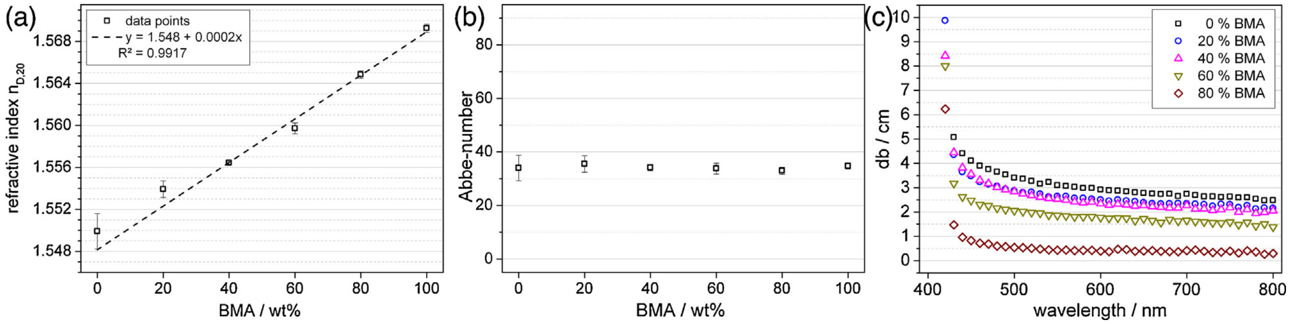

Fig. 2. (a) Refractive index of the examined material system dependent on BMA content. (b) Abbe numbers dependent on BMA content, calculated with refractive indices measured at 450, 589, and 680 nm. (c) Optical damping calculated from UV–vis measurements and corrected by thickness and the reflection losses occurring at the two interfaces of the respective sample.

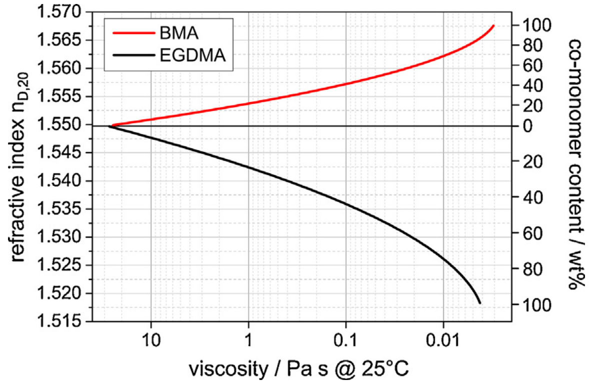

Fig. 3. Combined data of refractive index, viscosity and composition for the two material systems Syntholux and EGDMA and Syntholux and BMA, which show the achievable Δn for respective viscosities.

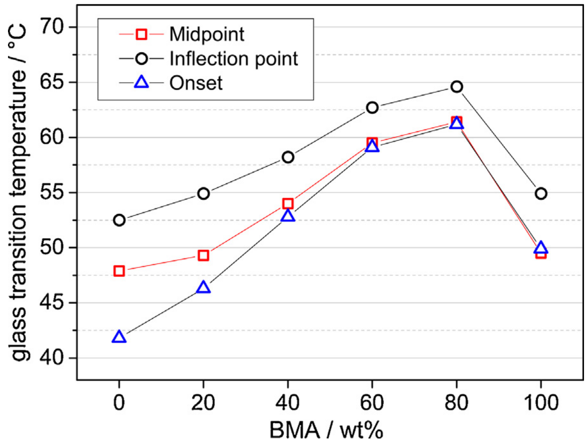

Fig. 4. Glass transition temperatures of the material system Syntholux and BMA.

polymerized samples of pure Syntholux show the lowest glass transition temperature of 52.5 °C, which then rises with increasing BMA content up to 64.5 °C for 80 wt% (Fig. 4). Pure BMA shows a lower glass transition temperature of only 55 °C which is caused by the lack of di-functionality of Syntholux and, thus, less available crosslinking leading to lower thermal stability.

4. Applications

The presented material system was used to fabricate different optical applications like rib waveguides for a Mach-Zehnder Interferometers (MZI), self-written waveguides (SWW), and for functional 3D printed structures.

4.1. Fabrication of inverted rib waveguides

At first the design and fabrication of rib waveguides on which Mach-Zehnder interferometers (MZI) are based on are presented. These kind of optical sensors probe a gaseous or liquid analyte by the evanescent field of the guided mode. In comparison to state of the art MZI using subtractive fabrication techniques, we present asymmetric rib waveguides. The advantages are avoiding of an interaction window and the usage of a homogeneous layer, which enables low cost and fast additive fabrication technologies like spin-coating or printing.

The rib waveguides are fabricated by using microsystems technologies. Therefore a silicon wafer was patterned using lithography and etching using a photoresist and nitride layer as mask. These structures are copied into a negative form by casting PDMS and afterwards using the PDMS as soft stamp to transfer these structures in a 175 μm thick PMMA bottom cladding foil by hot embossing. Subsequently, the hot-embossed grooves are filled by the core material.

The sensitivity S is an important parameter of the MZI system and depends on the whole system design, but mainly on the height of the waveguide core layers. In general, small heights are required to increase the sensitivity as well as the refractive index. For this application, the refractive index of the core layer for best sensor performance was tailored by suitable mixtures of Syntholux and EGDMA. A mixture of 40 wt% Syntholux, 60 wt% EGDMA and DPO as photo initiator was chosen, due to its viscosity for spin coating and cross-linking behavior. A 3 μm thick layer of this core layer is spin-coated onto the foil at 1800 rpm and cross-linked by UV illumination at 405 nm under a nitrogen atmosphere. This material

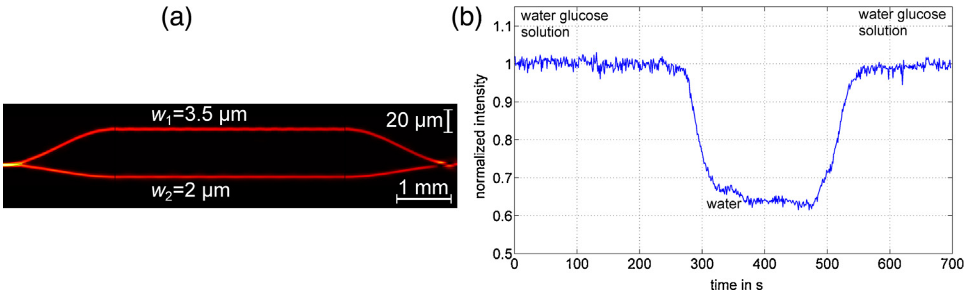

Fig. 5. (a) Top view of the designed asymmetric Mach-Zehnder interferometer with different waveguide width w in the two interferometer arms (different scales in x and y), (b) normalized measurement signal of the MZI when water glucose solution, water and water glucose solution is applied as upper cladding, respectively. The refractive index step is approximately 0.04.

composition results in a refractive index ng = 1.531 at 589 nm and a dynamic viscosity of 0.03 Pa·s. The tunability of the viscosity also enables the fabrication of the sensor structures by using printing techniques.

Fig. 5 (a) shows a top view of the designed asymmetric MZI with different waveguide width in the two interferometer arms. The system allows the measurement of refractive index changes with a limit of detection of 0.003 refractive index units [28]. Fig. 5 (b) shows a normalized measurement signal of the MZI when water glucose and pure water solutions are applied, respectively. The refractive index step between the different solutions is approximately 0.04.

4.2. *Self-written optical waveguides fabrication*

Self-written waveguides (SWW) are light guiding structures which are fabricated by guiding a laser beam through a liquid monomer leading to a rigid polymerized waveguide core [29]. The creation of SWW is based on a local increase of the refractive index due to exposure to UV light, which starts a local polymerization process. After an initial seed point in the material is polymerized, this part forms a light guiding structure leading to the polymerization of adjacent material in beam direction. Hence, an optical waveguide is formed in the monomer during UV light exposure. Application examples for SWW include, e.g., low loss optical interconnects to link optical fibers, waveguides, sensors or other optical elements [29]. However, to fabricate an SWW core but also a rigid cladding using only a single monomer solution requires the adaption of the material properties such as viscosity and refractive index to match the special requirements of the SWW process. Utilizing the material concept presented in this work, an SWW fiber-to-fiber interconnect including a rigid core and cladding was created [29]. For fabrication pure Syntholux was chosen as monomer due to its high viscosity which keeps the material at the right spot. Irgacure 184 was used as photo initiator due to its high reactivity with UV light. The material was then applied to a gap between two optical fibers. The first fiber was attached to a diode laser (MCLS1, Thorlabs, Newton, USA) with a center wavelength of 405 nm and acts as launching fiber. After exposure to the laser radiation, an SWW was formed inside the mixture which links both fibers, as shown in Fig. 6.

In the literature, SWWs were also reported by other groups [30–32]. However, in contrast to that work, we were able to establish a rigid waveguide cladding around the core by only one additional UV flood exposure process. Commonly, for creation of a rigid waveguide cladding, removing the remaining liquid monomer after core formation and exchanging it with another material is required to obtain suitable refractive index profiles for the light guiding structure. Using the presented material mixture is beneficial as the remaining liquid monomer surrounding the waveguide core can be polymerized with a different wavelength and then used as cladding. In contrast to previous work where a resin mixture which is curable at different wavelengths was used to generate a refractive index difference Δn between core and cladding, the Syntholux mixture yields Δn = 0.07 which is four to seven times larger than reported elsewhere [33,34]. For comparison, a refractive index profile of the fabricated SWW interconnect which was measured using the refracted near field method and a refractive index profiler [35] is shown in Fig. 7.

4.3. *Functional 3D structures using stereolithography*

The presented material system was also used to fabricate optical structures using stereolithography. Different material compositions with varying co-monomer ratios were used to create test structures using a 3D stereolithographic printer (B9Creator, B9Creations, USA). The effect of the co-monomer content on the quality of the test structures was optically analyzed (SZ61 stereo microscope, Olympus).

The different mixtures were prepared using co-monomer ratios of 20, 40, 60 and 80 wt% BMA and with 1 wt% DPO as the photo initiator. The test structures consisted of a Siemens circle and linear stripes of different widths, all with heights of 1 mm, printed at a voxel size of 50 × 50 × 50 µm at an illumination time of 3 s per layer. Post-processing steps included 5 min in an isopropanol US-bath to remove excess monomer on the surface of the structure, followed by drying and flood illumination at 405 nm.

Fig. 8 illustrates the effect of higher BMA content on the print quality. At 20 wt% BMA (Fig. 8 (a) and (b)), features as small as 100 × 100 µm could be realized. Higher co-monomer content led to a decrease in the structure quality (Fig. 8 (c) and (d)), with the features becoming rounder and features below 200 × 200 µm being blurred. Moreover, structures with higher BMA content were found to be thinner and more flexible. The optical quality as well as the mechanical flexibility of the structures printed with higher BMA is due to its lower curing capabilities in the presence of DPO. BMA can therefore be used as a modifier additive at less than 50 wt%, to alter the mechanical properties of the resulting structures.

5. Summary & outlook

In this work, we report on a novel host-guest material based on epoxy acrylate and suited for the realization of micro-optical elements and systems. The influence selected co-monomers introduced into the matrix material Syntholux was analyzed with respect to the rheological, optical and thermomechanical properties. With the described material system and fabrication method

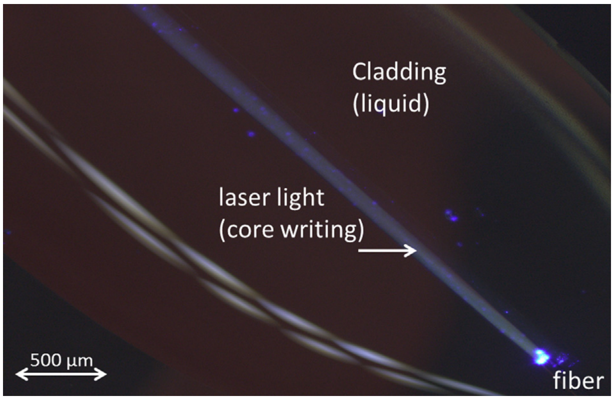

Fig. 6. Self-written waveguide (SWW) interconnect linking two optical fibers.

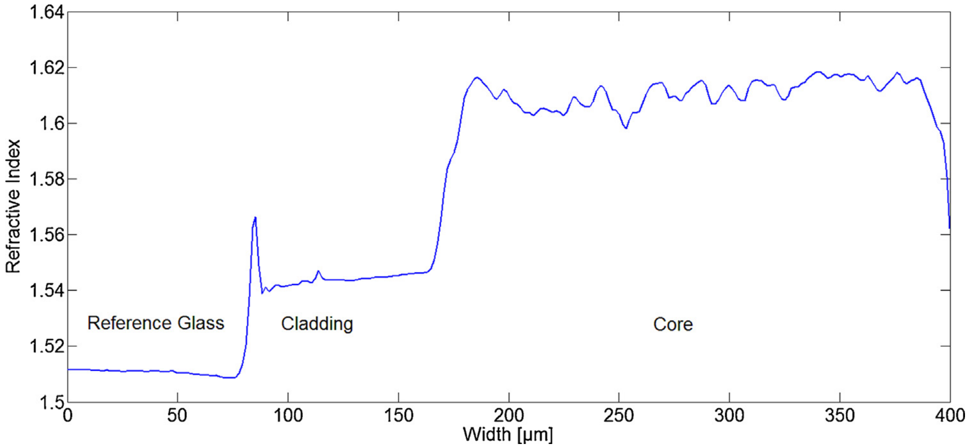

Fig. 7. Refractive index difference between core and cladding [29].

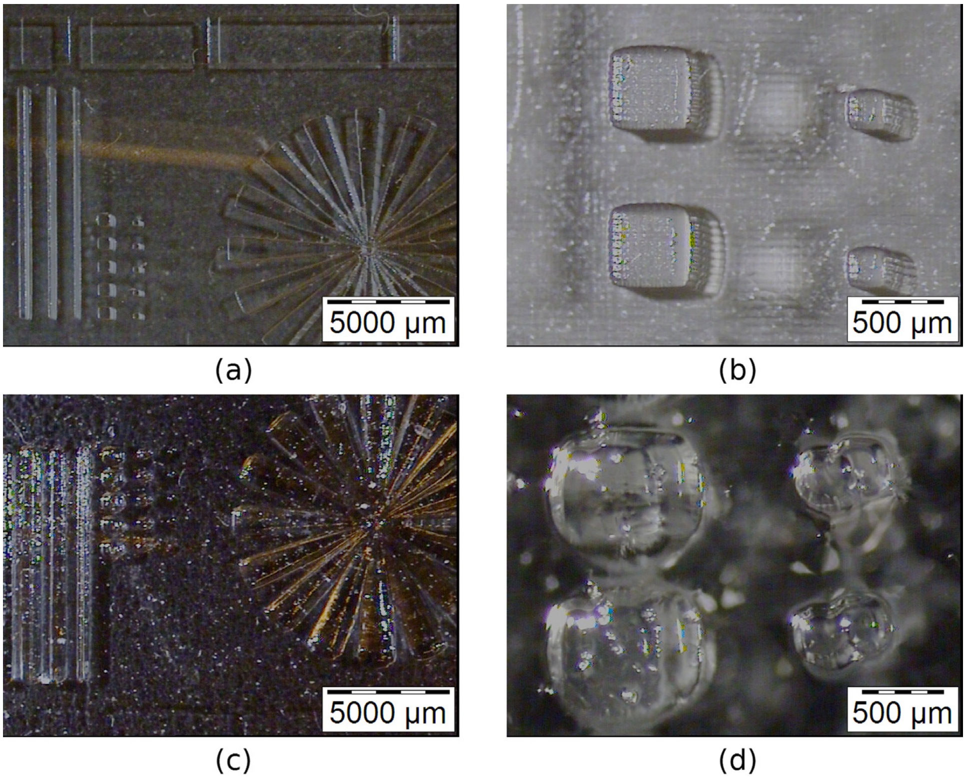

Fig. 8. Test structures printed using BMA ratios of 20 vol.% ((a) and (b)) and 80 vol.% ((c) and (d)). The structures with lower BMA content ((a) and (b)) are sharper with structure resolutions down to 100 × 100 µm × 1 mm. Higher BMA content led to reduction in the quality of the structures ((c) and (d)).

the viscosity of the material can be adjusted in a broad range between 0.003 Pa s and 46 Pa s by adding BMA or EGDMA. This way the material mixtures can be used by various microstructuring methods like spin coating, hot embossing or stereolithography to realize optical waveguide structures.

The refractive index of the material system can be adjusted between $n_{D,20} = 1.518$ and 1.569. A refractive index change Δn of up to 0.05 compared to the matrix material was achieved while the viscosity was kept constant. This matches the requirements for realization of optical waveguides using various production methods. One of the next steps will be the combination of BMA and EGDMA together with Syntholux. This will strongly enhance the independency of the refractive index from viscosity, leading to a completely variable material system. It appears feasible, to even further increase the refractive index by adding dopants, e.g. 9-bromo-phenanthrene or 9-vinylcarbazole.

The functional use of the presented material systems was then demonstrated with three example optical applications. Inverted rib waveguides were realized by filling a hot embossed PMMA substrate with the presented material. The surface roughness of the resulting waveguides was measured to be below 15 nm for a 3 μm thick core layer with $n_g = 1.531$ at 589 nm. The influence of the core height was shown to be relevant for the fabrication of Mach Zehnder interferometers.

Additionally, self-written waveguides were demonstrated by polymerizing the material using laser radiation at 406 nm. The refractive index difference between the polymerized core and cladding was around $\Delta n = 0.07$ which is sufficient to realize efficient optical waveguides. Finally, we showed that the material can be printed into functional 3D structures using stereolithography with reliable feature sizes down to 100 μm. In future, the material system developed will be used to realize novel planar-optical sensor systems based entirely on polymer materials.

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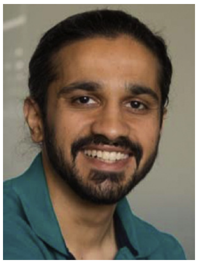
Biographies

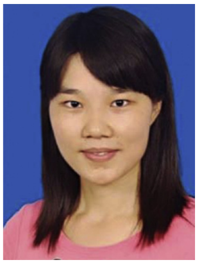
Uwe Gleißner received his BSc and MSc degrees in microsystems engineering from the Department of Microsystems Engineering, University of Freiburg, Germany, in 2009 and 2012, respectively. He is currently working at the University of Freiburg within the collaborative research center Transregio 123–Planar Optronic Systems (PlanOS), which is supported by the German Research Foundation. His current research is focusing on the development of optically and rheologically tailored polymers for integrated optics.



Bilal Khatri was born in Karachi, Pakistan in January 1988. After finishing his bachelors in electronic engineering from Iqra University, Karachi, he moved to Freiburg, Germany for his masters in microsystems engineering. His master dissertation involved the thermomechanical characterization of liquid crystal elastomers, a biomimetic material intended for use as an artificial muscle. Currently he is working towards his Ph.D. that involves structuring of functional material composites based on polymer matrices using fused deposition modelling and stereolithography.

Christof Megnin received his diploma and doctorate degree in mechanical engineering from the Karlsruhe Institute of Technology (Germany) in 2008 and 2013, respectivley. During his Ph.D. he worked at the Institute of Microstructure Technology on the development of microfluidic devices for integration in fluidic control systems. He is now working as group leader at the Laboratory for Material Process Technology at the Department of Microsystem Engineering of the University of Freiburg (Germany).

Stanislav Sherman studied microsystems engineering with the focus on life sciences at the University of Freiburg. He completed his academic master's degree with the master's thesis: "Development of a technology platform for optical measurement of vessel wall dynamics". 2013 he took up his position as a scientific assistant and a Ph.D. student at the Gisela and Erwin Sick Chair of Micro-optics in Freiburg, as a part of the Collaborative Research Center SFB/TRR 123 "Planar Optronic Systems – PlanOS". His current research is focusing on the development and integration of optical temperature sensors into polymer-based waveguides on flexible substrates.



Since September 2013, **Yanfen Xiao** joined the Gisela and Erwin Sick Chair of Micro-optics leaded by Prof. Hans Zappe at Albert-Ludwigs-Universität Freiburg (Germany), Department of Microsystem Engineering. From 2009–2012, she finished her master thesis in Shanghai Institute of Optics and Fine Mechanics, Chinese Academy of Sciences, China. Her recent study area refers to simulations and characterization of interferometric sensors fabricated on flexible polymer foils using hot-embossing and printing technology.

Meike Hofmann studied Micromechatronics at the Ilmenau University of Technology from 2000 to 2005. In 2006 she started working towards her Ph.D. at the department of Optical Engineering at the same university. Her activities covered research in the field of planar optical system integration and diffractive optics. In 2013 she received her Ph.D. and joined the Gisela and Erwin Sick Chair of Micro-optics at the University of Freiburg. Now she is working on the development of all-polymer waveguide based interferometers for gas analytics in foils.

Axel Günther received his B.Eng. from the University of Applied Sciences in Zwickau in 2011. Afterwards he studies the masters course Optical Technologies at the Leibniz Universität Hannover and finished with a M.Sc. degree in 2013. Currently, he is a Ph.D. student at the Hannover Centre for Optical Technologies. His research interests include polymer based micro optics, optical technology for life sciences and biophotonics.

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Hans Zappe was born in Paris and raised in New York. He earned Bachelor's and Master's degrees at MIT in 1983 and a Ph.D. from the University of California, Berkeley, in 1989, all in Electrical Engineering. He has worked at IBM (USA), the Fraunhofer Institute for Applied Solid State Physics (Germany) and the Centre Suisse d'Electronique et de Microtechnique (Switzerland). He joined the University of Freiburg in 2000, where he is now the Gisela and Erwin Sick Professor of Micro-optics in the Department of Microsystems Engineering.

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