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Christof Megnin
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Thomas Hanemann

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Ink-jet printed fluorescent materials as light sources for planar optical waveguides on polymer foils

Patrick Bollgruen,^{a,b,*} Uwe Gleissner,^b Tim Wolfer,^c Christof Megnin,^b Dario Mager,^a Ludger Overmeyer,^c Jan G. Korvink,^a and Thomas Hanemann^{b,d}

^aKarlsruhe Institute of Technology (KIT), Institute of Microstructure Technology (IMT), Hermann-von-Helmholtz-Platz 1, Eggenstein-Leopoldshafen 76344, Germany

^bUniversity of Freiburg, Department of Microsystems Engineering (IMTEK), Laboratory for Materials Processing, Georges-Koehler-Allee 102, Freiburg 79110, Germany

^cUniversity of Hanover, Institute of Transport and Automation Technology (ITA), An der Universitaet 2, Garbsen 30823, Germany

^dKarlsruhe Institute of Technology (KIT), Institute for Applied Materials (IAM), Hermann-von-Helmholtz-Platz 1, Eggenstein-Leopoldshafen 76344, Germany

Abstract. Polymer-based optical sensor networks on foils (planar optronic systems) are a promising research field, but it can be challenging to supply them with light. We present a solvent-free, ink-jet printable material system with optically active substances to create planar light sources for these networks. The ink is based on a UV-curable monomer, the fluorescent agents are $\text{Eu}(\text{DBM})_3\text{Phen}$ or 9,10-diphenylanthracene, which fluoresce at 612 or 430 nm, respectively. We demonstrate the application as light source by printing a small area of fluorescent material on an optical waveguide fabricated by flexographic printing on PMMA foil, resulting in a simple polymer-optical device fabricated entirely by additive deposition techniques. When excited by a 405-nm laser of 10 mW, the emitted light couples into the waveguide and appears at the end of the waveguide. In comparison to conventional light sources, the intensity is weak but could be detected with a photodiode power sensor. In return, the concept has the advantage of being completely independent of any electrical elements or external cable connections. © 2016 Society of Photo-Optical Instrumentation Engineers (SPIE) [DOI: 10.1117/1.OE.55.10.107107]

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1 Introduction

Planar optronic systems on foils manufactured by roll-to-roll compatible additive manufacturing techniques such as ink-jet or flexographic printing are a promising type of sensor network.¹ In comparison to conventional electronics, these systems are potentially lighter, cheaper, and faster. Additionally, they could be employed in high-voltage or high-magnetic-field environments where electrical equipment is difficult to handle, such as magnetic resonance (MR) scanners. Current research efforts aim toward the realization of a complete optical sensor system consisting of light sources, optical waveguides, embedded sensor structures, and light detectors on a flexible substrate. The sensor structures, designed to react to external physical properties such as temperature,² humidity,^{3,4} or elongation,⁵ alter the passing light in the waveguide so that it is possible to quantify a change of the corresponding physical property. By using networks of these systems, a large area can be monitored entirely by optical means, completely without electrical components.

As light sources, commercially available silicon-based light-emitting diodes (LEDs) and laser diodes can be used,⁶ but require a separate and time-consuming pick-and-place integration step, which hampers the mass fabrication by roll-to-roll. Additionally, they have a very high energy density, which poses a challenge for thermal management, especially on polymer substrates. Printed organic light-emitting diodes require several stacked materials and thereby increase the

number of manufacturing steps.⁷ Additionally, both these solutions rely on electric contacts to create the light, which defies the concept of a polymer-based all-optical and autonomous system.

To make the system independent from cables, one has to couple light from external optical sources through air. Typically, this requires precise alignment of the optical components and is very sensitive to vibrations or movement. We found that using a fluorescent material on the waveguides as an intermediate stage between a laser beam and the optical system leads to a much more robust coupling behavior. This enables autonomous planar optronic systems that do not rely on electrical power, as laser beams to excite the fluorescence within the system have long range and can even track and follow a moving system.

Ink-jet printing was chosen as a manufacturing technique because it allows a locally defined and contact-free deposition of the fluorescent light sources.⁸ Without any masking steps to be considered, structures with lateral feature sizes below 100 μm can be deposited on many different types of planar waveguides and substrates with minimal consumption of material.

By way of example, two fluorescent agents for such light sources were investigated. The first material is the europium-complex $\text{Eu}(\text{DBM})_3\text{Phen}$, which fluoresces red at 612 nm. As a counter part in the blue spectrum, the common fluorophore 9,10-diphenylanthracene (9,10-DPA) was selected, which emits light at 430 nm. Both materials are well known for their

*Address all correspondence to: Patrick Bollgruen, E-mail: patrick.bollgruen@imtek.uni-freiburg.de

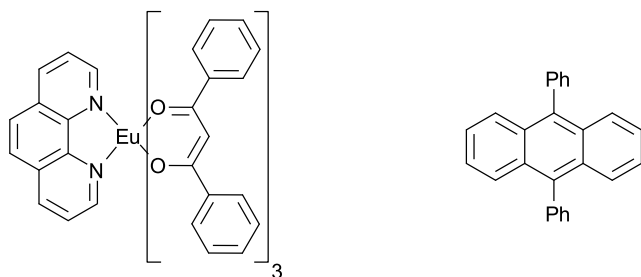


Fig. 1 Dopants used to achieve fluorescence. The left material was used for the europium-ink and the right material for the anthracene-ink. The fluorescence occurs around 612 and 430 nm, respectively.

suitability in fluorescent optical waveguides,^{9–11} but this is the first time that their deposition in a solvent-free polymer matrix via ink-jet printing has been reported. Other fluorophores could be deposited in a similar manner.

2 Materials and Methods

2.1 Ink Formulation

The inks are made from four components: an optically transparent base material, an additive to tune the viscosity for ink-jet printing, a photoinitiator that allows radical polymerization under UV-light, and the fluorescent dopants. No volatile solvents are required. Following previous work,¹² bisphenol A ethoxylate diacrylate (BE) was used as the base material. To adjust the viscosity, ethylene glycol dimethacrylate (EGDMA) was added in a ratio of 1:1, which leads to a viscosity of 10 mPa s when heated to 50°C. EGDMA as a viscosity reducing agent has the advantage that it polymerizes together with the base material, and does not vaporize as do common solvents, hence keeping the dispensed mass constant. As a photo initiator, diphenyl(2,4,6-trimethylbenzoyl)phosphine oxide (DPO) was added with 3 wt. %. To achieve fluorescence, two different dopants, as shown in Fig. 1, were added: an europium-based rare-earth complex [Eu(DBM)₃ Ph], with 2.5 wt. %, and 9,10-diphenylanthracene, with 0.5 wt. %. At these concentrations, the dopants did not interfere with the printability. The inks will be referenced to as europium-ink and anthracene-ink.

2.2 Ink-Jet Printing

Ink-jet printing is a technique where droplets with a volume between 1 nl and 1 pl are ejected from a print-head nozzle onto a substrate.¹³ While there are several methods to create droplets, the most common in laboratory use is the piezo-based print-head. Here, microfluidic channels with a diameter between 10 and 100 μm in a silicon chip are filled with ink. One end of the channel is open to air, forming a printing nozzle. Piezo actuators placed in the vicinity of the nozzle and actuated by a defined voltage pulse pattern cause an ejection of droplets. The print-head requires the ink viscosity to match a certain value, typically between 10 and 100 mPa s.

When the ink hits the substrate, the contact angle between the liquid and the solid surface defines the droplet shape, and ultimately, the minimal feature size. If the contact angle is high, the ink does not wet the substrate, and shows a phenomenon commonly known as “Lotus effect.” Other phenomena, such as solvent evaporation, drying, or ink pinning, can cause a wide range of morphological effects, which can be exploited to achieve the desired shape.¹⁴ After printing, the deposited material turns solid. For particle-based inks, this usually happens by thermal evaporation of the solvent. For polymer-based inks, such as in the current case, UV-initiated polymerization is most commonly used.

3 Experimental and Characterization

3.1 Ink

The materials were mixed under ambient conditions with a high-speed mixer (Ultra-Turrax, IKA, Germany). Mixing was performed for 1 min to achieve homogeneity. Air bubbles were removed by ultrasonic treatment performed for 5 min.

To measure the viscosity, a cone and plate rheometer (CVO50, Bohlin Instruments, Germany) with two different setups was used. First, temperature was increased linearly from 20 to 80°C at a constant shear of 200 s⁻¹. Second, shear rate was increased from 10 to 200 s⁻¹ at four different temperatures within this temperature window. The results, as shown in Fig. 2, show Newtonian behavior at all examined temperatures and viscosity ranging from 5 to 30 mPa s, which can be used for tuning the droplet formation when

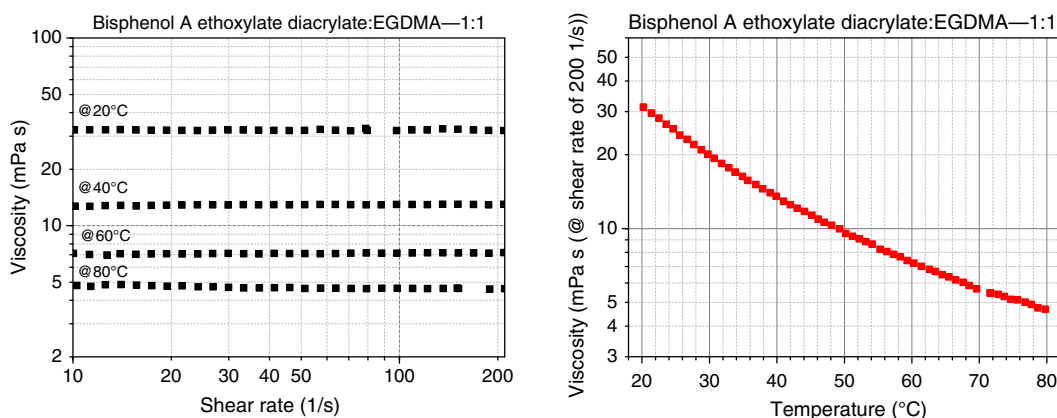


Fig. 2 Rheological characterization showing the ink viscosity (a) over shear rate at four constant temperatures and (b) over temperature at a constant shear rate of 200 s⁻¹. The ink shows Newtonian behavior at the investigated shear rates.

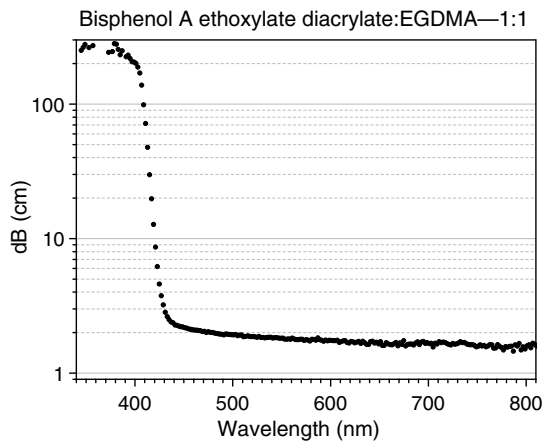


Fig. 3 Optical damping behavior of the polymerized ink without fluorescent dopants, measured with a UV-Vis spectrometer. The ink is transparent above 430 nm, which means that the embedded fluorophores are only UV-excited in relatively thin structures.

ink-jet printing. The rheological properties were measured on ink without the fluorescent dopants.

Similarly to the rheological characterization, the refractive index measurements were performed without fluorescent dopants. Polymerized samples were produced using a silicone mold between two glass plates. The ink was polymerized at 405 nm for 8 min with a radiation power of 0.25 W/cm^2 (LED Spot 100, Dr. Hönle AG, Germany). The refractive index of the liquid ink, as well as of the polymerized material, was measured at three different wavelengths (450, 589, and 680 nm) and 20°C using an Abbe refractometer (DR-M2/1550, ATAGO, Japan). This way, Abbe numbers could be calculated.

The results of this optical characterization show a refractive index n_D^{20} of 1.500 for the liquid ink. After polymerization, the refractive index increases to 1.545, with an Abbe number of 35.

The optical attenuation of the base material without dopants was determined with a spectrophotometer (Cary 50 UV-Vis, Agilent Technologies) between 400 and 800 nm. The reflection losses at the front and back interface due to the different refractive indices were calculated using the Fresnel equation. Then transmission with and without the

sample was measured and the optical damping was determined to be 1.5 dB/cm at 800 nm and 2.0 dB/cm at 450 nm. For shorter wavelengths, where the fluorophores are excited, the attenuation is significantly above 100 dB/cm . The results of this characterization are shown in Fig. 3.

To measure the emission spectrum of the fluorescent material, samples with fluorescent agents were placed in a PerkinElmer LS 55 fluorescence spectrometer (PerkinElmer). Here, light with wavelengths between 350 and 380 nm is used to excite fluorescence. The results of this characterization are shown in Fig. 4.

3.2 Printing of Fluorescent Structures

The inks were printed with a Dimatix DMP 2831 ink-jet printer equipped with a Dimatix DMC-11610 print-head (FujiFilm Dimatix), which deposits droplets of a nominal volume of 10 pl from up to 16 nozzles. The device was furnished with UV-intransparent foil to protect the UV-sensitive ink from ambient light. To tune the ink viscosity to a printable regime of 10 mPa s , the print-head temperature was set to 50°C for both inks.

Stable droplet generation was already achieved at a pulse voltage of 20 V, but was increased to 25 V for improved reliability and larger deposited volume. The substrate was $250\text{-}\mu\text{m}$ thick PMMA (99524 GT) foil. With these parameters and a drop spacing of $25 \mu\text{m}$, shapes with a minimum feature size in the range of $100 \mu\text{m}$ and a minimal thickness of $10 \mu\text{m}$ could be deposited. When not in use, the filled cartridge was stored at 8°C . The reliability was remarkably good, and the filled cartridge would print even after months of storage. Also, the material consumption was very low, as only one cartridge of 2 ml per ink was required for all experiments and samples. A printed sample with both fluorescent agents is shown in Fig. 5.

To measure the emitted intensity, samples with 3 and 9 layers of 400×400 droplets were printed, resulting in squares with a side length of 10 mm. After printing, the ink was exposed to UV radiation with an irradiance of 1.5 W/cm^2 at 365 nm (FireFly 25×10 , Phoseon Technology) for 30 s to initiate radical polymerization. To prevent oxygen inhibiting this process, the ambient air at the sample was flushed away with nitrogen to an oxygen concentration below 0.5%. The thickness of the printed structures,

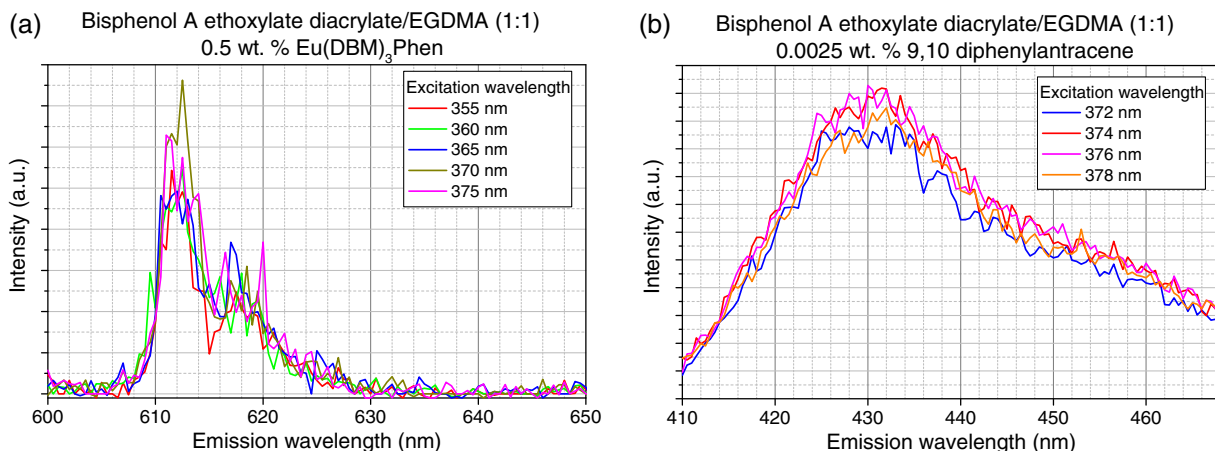


Fig. 4 Fluorescence spectrum of (a) the europium-ink and (b) the anthracene-ink, which shows that the emission peak does not depend on the excitation wavelength.

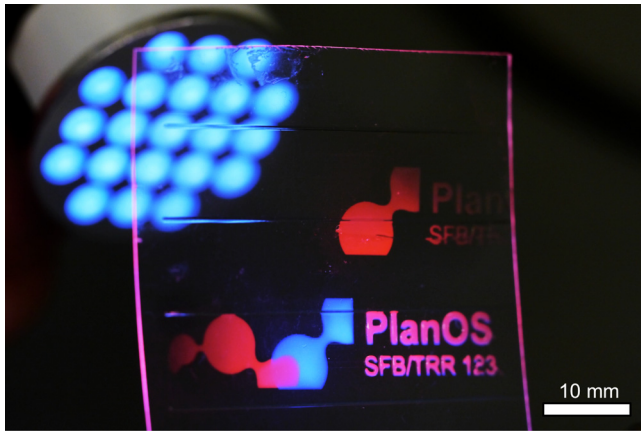


Fig. 5 Test structure printed on foil, giving an impression of the printed material under UV. Areas that appear pink were covered by both inks.

measured with a digital caliper, was about $13 \mu\text{m}$ per layer. Thinner structures could be realized by reducing the number of printed layers, a lower pulse voltage, or increasing the droplet spacing, but at the cost of intensity.

The samples were placed on a silicon-based photo diode (S151C, Thorlabs) with the fluorescent area facing up. The optical excitation was performed with an intensity of $100 \text{ mW}/\text{cm}^2$, at 405 nm for the europium-ink, and at 365 nm for the anthracene-ink (LED Spot 100, Dr. Hönle AG, Germany). In the case of the europium-ink, an optical long-pass filter with cut-on at 450 nm (FEL0450, Thorlabs) was necessary to filter the excitation light. For the anthracene-ink, the PMMA substrate acted as filter. To rule out any contribution to the measured signal from other sources, a differential measurement with a clear foil substrate was performed. The results of the characterization are shown in Fig. 6. The characterization showed a continuous dimming for both fluorophores. The effect lessened over time. After 5 min, the intensity dropped at a rate between 1 and 5%/min. Although the fluorophores recovered some of their intensity, partial permanent bleaching was also observed.

4 Application

To investigate the suitability of the presented materials for the application as light sources, foil-based polymer-optical

waveguides were furnished with fluorescent elements, leading to a simple polymer-optical device fabricated entirely by additive deposition techniques. The waveguides were fabricated by flexographic printing of a transparent acrylic varnish on PMMA foil.¹⁵ The waveguide shape is a circular arc 0.7-mm wide and 0.1-mm high, which results in a radius of curvature of 0.7 mm and a cross-sectional area of 0.05 mm^2 . The refractive index of the polymerized waveguide material is 1.516 at 635 nm , the surface roughness is below 50 nm , and waveguide losses were measured to be below $0.5 \text{ dB}/\text{cm}$ at this wavelength.

A fluorescent area of 0.49 mm^2 was printed in three layers on an 80-mm long waveguide at one end. To absorb any stray light, the backside of the waveguide foil was sprayed black. The end facet was polished on rotating abrasive sheets with grit sizes P320, P800, and P2400 at 150 rpm for 30 s each (Phoenix Beta, Buehler, Germany). By pointing a collimated 405-nm laser beam of 10 mW (S1FC405, Thorlabs) and an approximate spot size of 1 mm on the fluorescent area, light could be excited within the waveguide from more than a meter away without any embedded coupling structures such as gratings or nanoparticles. Alignment by hand was sufficient to align the beam with the light source and achieve light in the waveguide. In case of the europium-ink, the 612 nm light was well visible at the facet. For the anthracene-ink, the facet remained dark, which indicates that the attenuation in the flexo-printed waveguides at 430 nm is very high. The device with europium-ink is shown in Fig. 7.

To measure the intensity at the end facet, the photodiode from the previous measurements was furnished with a 1-mm aperture and a 450-nm long-pass filter to absorb the blue laser light and was placed directly at the facet. After bridging the gap between the waveguide end facet and the detector with immersion oil, the photodiode recorded a power difference of 28 nW upon activation of the excitation laser.

5 Discussion

The power collected at the end facet of the demonstrator by the photodetector lies in the nanowatt range, which poses a challenge for signal detection. First, it should be stressed that the gap between facet and detector, although bridged with immersion oil, causes significant losses, and that the light was bright enough for both the bare eye and a standard CMOS sensor. Still, the measured value is given to allow

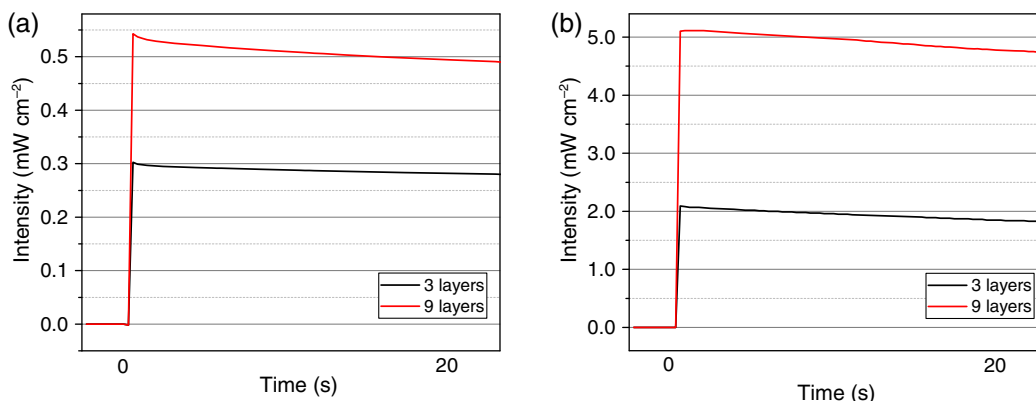


Fig. 6 Measured intensity of printed squares with 3 and 9 layers, (a) europium-ink and (b) anthracene-ink. Generally, anthracene-ink shows an intensity about 1 order of magnitude higher than europium-ink. Nine layers are only about twice as bright as three layers due to UV-attenuation of the base material.

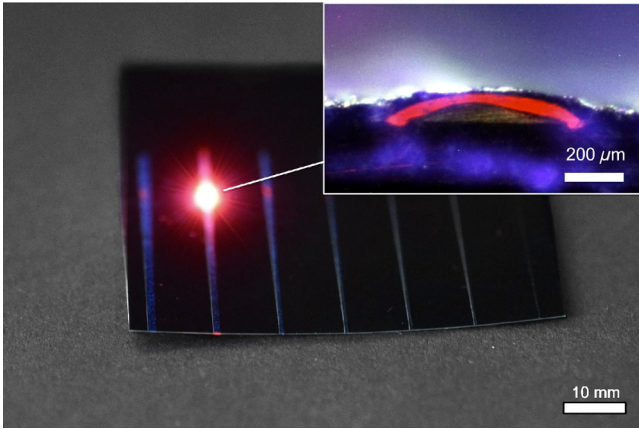


Fig. 7 Photograph of the application experiment showing a foil with 80-mm long flexographically printed optical waveguides and the printed light source and the end facet at the bottom foil edge glowing red. The inset shows the cross section of the printed light source, with the fluorescent material bright red covering the waveguide below.

a comparison with future work characterized in similar setups. Two factors contribute to the light intensity at the rear facet: the conversion efficiency and the coupling efficiency.

Figure 6 shows that three printed layers of europium-ink convert about 0.3% of the UV-light used for excitation. This yield could be improved by printing additional layers, but the effect soon wears out because the excitation light is absorbed by the host material. Still, values up to the one-digit percent range are realistic for this fluorophore. The second investigated material, anthracene-ink, shows a much higher intensity, which points to a better conversion efficiency, but was unsuitable for this particular type of acrylate-based printed waveguide because of high attenuation at this wavelength. Yet this points to the fact that other fluorophores at suitable wavelengths might offer higher intensities and could be used in a similar fashion as europium-ink.

Both inks showed a certain degree of photonic bleaching, which could be counteracted by using a longer wavelength or pulsed mode for excitation. Alternatively, fluorescent nanoparticles could be added and might work similarly well for fluorescent light sources, but with the typical disadvantages of ink-jet printed nanoparticles, such as nozzle clogging during printing and scattering due to agglomeration.

The second factor is the fraction between the light emitted toward the waveguide and the light picked up by the waveguide, commonly known as coupling efficiency. Currently, the refractive index of the ink is at 1.545, which is above the refractive index of the waveguide material (1.516). This causes part of the emitted light to reflect back into the fluorescent layer and eventually become absorbed there by the host material. The light which enters the waveguide needs to reflect at the bottom interface. With the refractive index at 1.49 for the PMMA substrate, the critical angle for total internal reflection is around 75 deg, which means that only light hitting the interface with an angle of 15 deg or less experiences total internal reflection. The majority of the light passes through the interface and is absorbed by the black substrate coating.

To improve the ratio between emitted and coupled light, one possibility is to investigate refractive index tuning as it was performed on other printable material systems.⁴ When

the printed waveguide core has the highest refractive index in the system, more of the light emitted in the fluorescent layer travels into the waveguide core. More sophisticated coupling structures, such as reflectors, gratings, or scattering nanoparticles, especially at the interface between waveguide and foil, would be a further option, but negate the simplicity of the presented concept and cannot be created by printing techniques alone.

6 Summary and Outlook

Ink-jet printed fluorescent polymer structures were successfully demonstrated as light sources on printed polymer-optical waveguides on foils. For the current ink formulation, the materials were very well printable without use of any volatile solvents, and no clogging was observed even after months. By having a tunable viscosity, the ink is not limited to a certain printer system, but can be adapted to any piezo-based print-head. Viscosity values outside the window of 5 to 30 mPa s can be reached by tuning the ratio of the ink components. The wavelengths emitted under UV illumination were 612 nm for the europium-ink and 430 nm for the anthracene-ink. Europium-ink was used to create a fluorescent light source on a flexographically printed polymer-optical waveguide, which led to a weak but visible light emission at the end facet of the waveguide.

In conclusion, the presented concept can be used to create light in polymer-optical waveguides and sensor systems. Although it offers lower intensities than conventional diode-based light sources, and needs to be integrated in a way that allows it to be targeted by a laser beam, the advantages are that it does not require electrical components or external connections and is polymer-based, thus, it is light and flexible, does not heat up on operation, and can be created with inexpensive mass-fabrication methods. The observed fading and bleaching of the fluorophores remain an open question, and has to be investigated if the structures should be used in long-term applications.

In the current state, the structures can be used for short time intervals, which is acceptable for disposable low-cost applications. Exemplarily, the system could be used to create light in optical sensor networks on rotating lab-on-a-disc^{16,17} and lab-on-a-foil¹⁸ systems, or in disposable optical sensor systems for the high-magnetic-field environment of an MR scanner.

Future activity is directed to the investigation of other fluorophores to cover a wider range of the light spectrum, as well as increased conversion efficiency. Additionally, refractive index tuning of the host material or embedded coupling structures is expected to result in higher light intensity in the waveguide.

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- Patrick Bollgruen** graduated with a MSc degree in microsystems engineering at the University of Freiburg in 2012 and worked in the fields of centrifugal microfluidics, terahertz metamaterials, and magnetic resonance imaging. Currently, he is working on optical waveguides fabricated by ink-jet printing of both passive and active polymer materials within the collaborative research center Transregio 123–Planar Optronic Systems (PlanOS), supported by the German Research Foundation. He got involved with printing under Dr. Patrick Smith.
- 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. Currently, he is working at the University of Freiburg within the collaborative research center Transregio 123–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.
- Tim Wolfer** received his MSc degree in mechanical engineering from Leibniz Universität Hannover in 2012. Currently, he is working as a research associate at Hannover Centre for Production Technology within the collaborative research center Transregio 123–PlanOS, which is supported by the German Research Foundation. His research interests are additive manufacturing of optical systems, computational fluid simulations of printing processes, and optoelectronic sensor systems.
- Christof Megnin** received his diploma and doctorate degree in mechanical engineering from Karlsruhe Institute of Technology (Germany) in 2008 and 2013, respectively. During his PhD he worked at the Institute of Microstructure Technology on the development of microfluidic control systems. He is now working as group leader at the Laboratory for Material Process Technology at the University of Freiburg (Germany) on developing of adjustable polymers and ceramics for additive fabrication.
- Dario Mager** studied MEMS technology at the University of Freiburg. His diploma thesis was about the simulation of pseudohall stress sensors in silicon. For his PhD thesis, he worked in the field of inkjet printing for micromanufacturing. In 2010, he became group leader for low-cost MEMS in Prof. Korvink's lab at the same university. In 2015, the group moved to the Karlsruhe Institute of Technology, where he is continuing his research.
- Ludger Overmeyer** received his doctorate degree in mechanical engineering at Leibniz Universität Hannover in 1996. He is a full professor and director of the Institute of Transport and Automation Technology at Leibniz Universität Hannover since 2002 and executive director of the Laser Zentrum Hannover since 2013. He is heading the collaborative research center Transregio 123 PlanOS and his research interests include transport and automation technology, optical production technologies, and laser material processing.
- Jan G. Korvink** is director of the Institute of Microstructure Technology at the Karlsruhe Institute of Technology and is in charge of the Helmholtz Research Programme "Science and Technology of Nanosystems." He is a fellow of the Royal Society of Chemistry, as well as a member of ASME and the IEEE. He was awarded an ERC Senior Grant for work in NMR metabolomics of *C. elegans*, and has coauthored around 150 journal papers.
- Thomas Hanemann** received his doctorate degree in physical chemistry from TU Darmstadt in 1993. He was a visiting scientist at IBM Almaden Research Center San José in 1994–1995. He was a head of the laboratory for materials processing at the Department of Microsystems Engineering at the University of Freiburg in 2011. His research topics include nonsilicon materials in microsystems technologies, polymer-nanomaterial composites, replication technology, and optical materials.