Research article

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Multifunctional materials for lean processing of waferscale optics

Abstract: The continuous miniaturization of components and devices along with the increasing need of sustainability in production requires materials which can fulfill the manifold requests concerning their functionality. From an industrial point of view emphasis is on cost reduction either for the materials, the processes, or for both, along with a facilitation of processing and a general reduction of resource consumption in manufacturing. Multifunctional nanoscale materials have been widely investigated due to their tunable material properties and their ability to fulfill the increasingly growing demands in miniaturization, ease of processes, low-cost manufacturing, scalability, reliability, and finally sustainability. A material class which fulfills these requirements and is suited for integrated or waferscale optics are inorganic–organic hybrid polymers such as ORMOCER®s [ORMOCER® is registered by the Fraunhofer Gesellschaft für Angewandte Forschung e.V. and commercialized by microresist technology GmbH under license since 2003]. The combination of chemically designed multifunctional low-cost materials with tunable optical properties is very attractive for (integrated) optical and waferscale applications via a variety of different nano- and microstructuring techniques to fabricate micro- and nano-optical components, typically within less than a handful of process steps. The influence of photoinitiator and cross-linking conditions onto the optical properties of an acrylate-based inorganic–organic hybrid polymer will be discussed, and its suitability for being applied in waferscale optics is demonstrated and discussed for miniaturized multi- and single channel imaging optics.

Keywords: hybrid polymers; lean processing; microoptics; multifunctionality; spectroscopy; waferscale.

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1 Introduction

Modern communication technologies require optical components which can be manufactured at low cost and which fulfill the increasingly growing demands in miniaturization [1–3]. This trend to miniaturization began several decades ago, and it is still an ongoing task, while at the same time their complexity and functionality of devices is continuously advancing. Integrated passive and active optical devices capable of waferscale processes are the key components in current and future data transfer technologies, and they have to be implemented in an environment with an excessive need for bandwidth increase at cost requirements of far less than 1 $/Gbit for the transceivers [4]. Not only data communication will benefit from integration and waferscale processes [5], but actually any technology of the 21st century such as mobile devices, augmented, mixed, and virtual reality (AR/MR/VR) [6], displays [6, 7], and quantum computing [8–10].

The tremendous demand on optical components has boosted the development of integrated optics and planar diffractive and refractive micro-optics on waferscale, and, driven by miniaturization and scaling, more and more ideas transformed into products for light shaping, guiding, and imaging at reasonable cost in the last decade. Examples are camera modules for smart phones [11, 12] or Köhler Integrators with [13] or without integration of illumination optics for automotive lighting [14], and displays [15]. In addition, there is a growing demand to develop large-volume, high precision production techniques for manufacturing [16], since a small-scale fabrication is time-consuming, expensive, and has higher hurdles to take to create uniformity and repeatability.

There are different techniques for the manufacturing of optics [17, 18] with many of them requiring multiple process

steps until the final optical element is achieved. The method which is used for the fabrication of the optics is determined by the application and its requirements on the optical element and the materials involved. The most common techniques for the fabrication of master tools are grinding and diamond turning, combined lithography/replication [3, 18], and Laser Processing including Direct Laser Writing (DLW) [19–24]. The master tools serve for mass production of polymer optical elements using injection molding [25], step-and-repeat replication [26], or roll-to-roll fabrication [27].

Standard or special glasses as well as polymer materials are the most common materials used in microoptics fabrication for waferscale or integrated optics fulfilling the high requirements of surface finish, shape accuracy, uniformity, and reproducibility. To fulfill future requirements of continuous miniaturization for refractive and diffractive optical elements, materials which enable device fabrication from the cm down to the nm scale with highest thermal stability and compatibility to processing techniques used in conventional semiconductor devices production have to be employed. While polymer materials are used for cheap optical elements or for fast prototyping of simple optical designs, inorganic–organic hybrid polymers such as ORMOCER®s are used for the production of waferscale optics due to their outstanding optical, thermal, and mechanical properties compared to standard polymer materials. These multifunctional nanomaterials are well-known in industrial environment, and they can be implemented in a vast variety of processes including semiconductor and printed-circuit board processing [1, 14, 28–30].

This publication is organized as follows. In a first section, a brief overview of general aspects of materials for optics and the material class of inorganic–organic hybrids are given, followed by a more comprehensive view onto a selected inorganic–organic material system for waferscale fabrication. The processing of the selected optical multifunctional material is discussed with respect to the material's properties and the underlying techniques, suitable to create waferscale optics with only a few process steps. Finally, selected applications examples are given.

2 Materials

2.1 Classical optical materials: glasses and polymers

For optical applications and products where microoptical elements are needed, materials which are transparent at the application wavelengths or wavelength bands are needed. Dependent on the community and the products which are realized, transparent inorganic glasses and organic polymers are still the most used materials for optical applications. These

materials might underlie some restrictions which have to be taken into account when using them for optical devices, resulting from their structure and/or their processing technologies. Thus, many of them are not suited for waferscale applications or need some effort to employ them on waferscale which comes along with high cost.

Glass processing is already established and continuously advancing over the last decades. Inorganic glasses are widely applied in discrete optical and opto-electrical components such as LEDs which have set a trend in lighting. When it comes to durability, glass is often the first choice for long-life products, although needs it is mechanically not flexible, highly brittle, and usually need high temperatures for processing. The latter restriction hinders, for example, its application in sequentially built-up technology on (large-area) polymer substrates for low-cost optics and opto-electronics with temperature-sensitive devices.

A very old method is shaping of glass via grinding and polishing where the optical element is first grinded close to its desired shape, followed by polishing the glasses surface to the final surface finish. This method is not practical for individual lenses with diameters of less than 1 mm or monolithic arrays of lenses [18]. Another method typically employed for the fabrication of all kind of discrete glass optical elements is glass molding (or glass blank pressing) [31], followed by many refinement steps to create the final optical element's design. Advantageous is that this process is very well-established over decades, that glass is a very reliable material, and that it works for a vast variety of products. The disadvantages when considering their implementation in waferscale processes are at hand. The formation of the optical elements is discrete, with elements smaller than 1 mm in diameter being hard to be fabricated. Diameters range from about 1 to 100 mm (Figure 1), and they can go down to about 400 μm for some special cases [31] by standard glass molding. The process needs very high temperatures up to 1300 °C dependent on the glass composition and its properties to soften the glass in the coarse shaping process. With ultra-precision glass molding [32], many process steps to achieve the final surface finish can be avoided, and the diameter of optical elements range between about 500 μm and 70 mm, with flank angles of less than 60° depending on the application. Dependent on the post-processing, surface roughness rms (root mean square) values are ranging from approximately 5 nm to 1.5 μm [33]. In addition, not all shapes which might be required by the application in particular regarding free-form optics, can be realized.

As for many techniques, suited tools have to be fabricated for the two methods, and the quality of the tool and its cleanliness determines the quality of the resulting optical elements as in any other fabrication method. Dependent on the designs, not all shapes can be realized, and the cost increases with the complexity of the tools and the needed processes.

Another fabrication method which can directly fabricate structures is diamond turning, which is used to fabricate tools or discrete optics in metals, glasses, and polymers [18] with diameters down to about 500 μm. These tools are then used for embossing of optical elements. Also, for diamond turning the quality of the tools and the subsequent post-processing determines the element's optical surface quality. Commercially available are high quality optical elements [34] with wavefront errors ranging from 1λ down to $\lambda/4$ (determined @632 nm) and surface roughnesses rms ranging from 15 nm to below 3 nm, with the resulting values dependent on the materials and the element sizes. For crystalline materials and polymers, rms values below 5 nm are achievable for substrate sizes up to 8″. For packaging,

Figure 1: Glass optics of 20–30 mm in diameter fabricated via glass molding. (a) Selection of different optical lenses and (b) variety of collimation optics. Courtesy of Schäfer Glas GmbH [31].

the discrete elements have to be assembled with the devices which requires additional (mainly) active alignment and gluing steps.

Discrete assembly processes are not suited for low-cost waferscale processing, and most of these technologies have not achieved the high level that wafer-based technologies provide, for example with respect to accuracy. Purely organic polymers can overcome some of the disadvantages of glasses, since they are mechanically flexible and can be processed at much lower temperature. The most commonly used optical polymers are polymethylenemethacrylate (PMMA), polystyrene (PS), and polycarbonate (PC) among others [35]. These materials are thermoplastic low-cost materials exhibiting a good processability which is typical for organic polymer materials. For example, optical components are built using hot embossing [36] or injection molding [25] at much lower temperature as compared with glass processing. However, purely organic polymers do not fulfill some major requirements if multilayer devices or multi-chip modules (MCM) have to be fabricated. They are often chemically not stable against solvents typically applied in multilayer and semiconductor processing, and their thermal stability is often low (80–120 °C for PMMA, depending on molecular weight). While PMMA shows high UV stability, PC has a much lower UV stability. In order to improve the UV stability, polycarbonate materials are either chemically stabilized or a UV absorber layer is deposited on top of the components via co-extrusion [37]. Compared to glass, the mechanical stability of organic polymers is lower. As for inorganic glasses, their refractive indices and other physical properties cannot be simply adapted to any application or processing technique.

2.2 Multifunctional materials for waferscale optics

The possibilities of nanotechnology to create multifunctional materials which can fulfill different functions in a device using only one material [29] seem to be infinitely, and tomorrow's products – although already small and smart – will become even smaller, smarter, and more

functional than ever before. Considering the unique properties of glasses and organic polymers, it is highly desirable to combine those to a material class combining the advantages of both worlds (Figure 2). This has resulted in the invention of the material class of inorganic–organic hybrid polymers about four decades ago [38–40] being nowadays well-established and commercially available in exceptional high quality. This material class is widely known under its brandname ORMOCER® [41] being derived from ORganically Modified CERamics, which describes the principle of combining inorganic and organic parts on a molecular level quite correct, but often is misleading in the sense of its temperature stability. While ceramics can be treated at temperatures far beyond 500 °C, this is not possible with ORMOCER®s if the inorganic *and* organic components should be maintained as integral part in the material. Nevertheless, compared to purely organic polymers hybrid polymers possess an increased temperature stability with some ORMOCER® materials being temperature-stable up to 400 °C, which was verified via thermogravimetry at an annealing rate of 5 K/min [42]. Most of the inorganic–organic hybrid polymers employed in microsystems technology are stable up to around 280–300 °C which is sufficiently high for subsequent processing such as soldering or metal layer deposition [30].

To combine the advantages of inorganic and organic materials, different syntheses routes can be used. The simplest one is to physically blend an organic polymer with inorganic particles which can be functionalized with an organic shell such that covalent bonds with the organic moieties of the polymer host material are formed [30, 42–44]. However, for an optical material with low haze very small inorganic particles ($d < 25$ nm) have to be employed, and their aggregation during storage and processing has to be avoided making the formulation and adaptation of such polymer-nanoparticle composites very difficult. However, suitable particle functionalization along with specialized know-how in composite preparation leads scattering-free optical materials upon processing [42].

A more sophisticated method to synthesize inorganic–organic hybrid materials which can be used in waferscale processing is the in-situ synthesis via a modified sol–gel process in one- or multiple steps [45–49]. The combination of inorganic and organic networks on a molecular level results in a material class where properties of organic polymers such as toughness, functionalization, and low-temperature processing like classical resist materials are combined with those of glassy materials such as hardness, chemical and thermal stability, and transparency [45]. Thus, inorganic–organic hybrid polymers such as ORMOCER®s are well-suited as integral part in industrial products, since they overcome the restrictions of purely organic polymers for most applications and show extremely high reliability determined by reliability testing [22].

The synthesis of inorganic–organic hybrid polymers is done via catalytically controlled hydrolysis and (poly)condensation reactions of organosilane precursors, and it is widely described in the literature [44–46, 50]. As mentioned above, the syntheses of ORMOCER®s which are used in microsystems technology to create waferscale or integrated optics, has to be modified compared to the standard sol–gel process. Classically, a sol–gel process consists of a hydrolysis and (poly) condensation reaction, where a sol is formed which then can be coated onto a substrate or a device. The remaining silanol groups $[SiOH]_n$ in the sol then further react, resulting in a gelation (solidification) along with a significant viscosity change of the material. To speed up this solidification, the process is accelerated by temperature or UV light exposure. Sol–gel coatings are applied in a vast variety of products, for
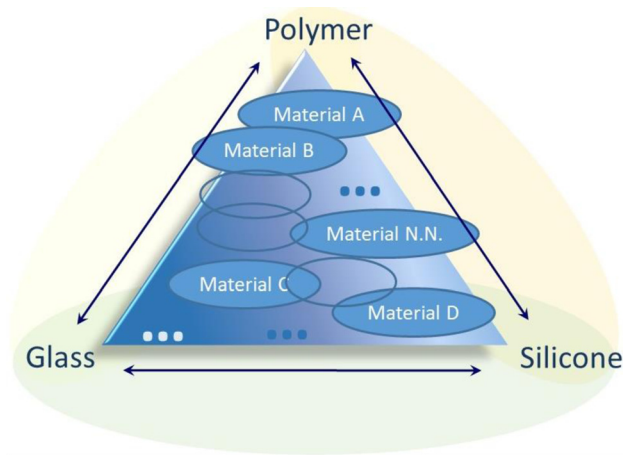
Figure 2: Schematics of the material class of inorganic–organic hybrid polymers, visualizing the infinite amounts of possible materials with different properties (Materials A – N.N.). Dependent on the synthesis conditions, the materials can be more glass-like, polymer-like, or silicone-like, resulting in the different properties. Dots and empty ellipsoids leave room for even more material ideas.

example in scratch-resistant layers for glasses and polymer lenses, in decorative layers, or in corrosion protective layers [51].

For microsystems technology, it is important that the resist material does not change its viscosity at least for half a year or even longer, since it is used as a *functional* resist material in conventional semiconductor processes (cf., section 3) where the processing conditions of the materials are required to be extremely stable to account for reliable processes and devices. Thus, it is advantageous to temporarily separate the sol–gel reaction during material synthesis and the solidification of the material during device fabrication. This is done by stopping the sol–gel reaction just before gelation of the material during synthesis resulting in a liquid and stable hybrid polymer resin, which can be supplied in a solvent-free form. During (waferscale) fabrication of the final components, solidification of the resin is achieved by crosslinking of the organic domain, i.e. temperature or UV-assisted curing of the photochemically reactive functional groups, for example (meth)acrylate, epoxide, or styryl moieties.

As for classical sol–gel synthesis, the synthesis typically starts with alkoxysilane precursors in a catalytically and thermally controlled reaction with or without extrinsic water [44, 46]. Aside of alkoxysilanes also heteroelements such as zirconium, aluminum, or titanium can be introduced into the inorganic network to further tune the material properties [52, 53]. These heteroelements may lead to higher refractive index material for waferscale applications or better scratch and abrasion resistance [51] compared to material modification without heteroelements. Additionally, the incorporation of heteroelements into the inorganic matrix via sol–gel processing leads to a better control of the material's homogeneity, since separation and agglomeration of structural elements as often observed with polymer-nanoparticle composites cannot occur. It has to be mentioned, however, that a significant increase of the inorganic content in the material reduces its capability to build thick layers due to increased brittleness, i.e. materials with high inorganic content usually cannot be processed in very thick layers of up to several 100 µm.

The profound knowledge about the underlying reactions and the detailed control of the precursor's quality as well as of the syntheses at any time is a pre-requisite for supplying high quality inorganic–organic hybrid polymers for waferscale and integrated optics applications. The synthesis conditions significantly influence the resulting material properties, starting with the choice of kind and/or the amount of alkoxysilane precursors, which build the basis for the material's chemical and physical properties. The way how the synthesis is carried out with respect to its conditions related to catalyst type and loading, solvents, and finally the reaction temperature determine the formation of the structural network elements on the nanometer scale. If one condition is changed, the resulting material might be changed completely in its properties, because a different inorganic-oxidic network is formed. This means that the material class of inorganic–organic hybrid materials such as ORMOCER®s is infinite in its number of possible multifunctional materials, and that the knowledge of how to control the syntheses is leading to customized and very reliable materials with well-defined property profiles. Since their physical and chemical properties can be tailored as required by the application and the processing technology, they can be employed in many devices for a large variety of optical, electronical, and opto-electronic (o/e) applications, particularly for waferscale and PCB processing [1, 28–30, 54–56]. The resulting materials are pre-polymer resins which consist of organically modified inorganic-oxidic networks with the individual parts covalently bonded on a molecular level [46, 50], and which are further processed to produce the optical element (cf., section 3).

Principally, each synthesis follows the same routine, but different considerations concerning the material's composition have to be carried out dependent on the application scenario. For example, if hybrid polymers will be used for single-mode waveguide fabrication [1], it is very important that the precursors are free of water and that the synthesized material is free of silanol groups, since –OH groups and water are known to show significant absorption at 1310 and 1550 nm [30, 57]. Also, the homogeneity of the material should be extremely high, and the structural features in the layers and structures should allow for nanometer surface finishes with a low rms value and a negligible shape deviation which is a pre-requisite for functional optical elements. Exemplarily, the homogeneity of a commercially available waveguide material (OrmoCore) processed on a Si(100) wafer was investigated by Timpel et al. [58] using focused ion beam preparation and high-resolution transmission electron microscopy, demonstrating that the material is extraordinarily homogeneous with structural features smaller than 1 nm which corresponds very well to SAXS (Small Angle X-Ray Scattering) measurements [59].

2.3 Spectroscopic characterization

Principal properties of inorganic–organic hybrid polymers were studied by means of spectroscopic investigations using the materials OrmoComp® and OrmoClear®, OrmoCore. OrmoComp® is a hybrid polymer obtained by a multi-step hydrolysis and polycondensation of an alkoxysilane precursor and the organic crosslinker TMPTA (trimethylolpropane triacrylate) in an aqueous environment. Its acrylate functionalities in the organic domain grant high reactivity and fast processing during UV curing of the material, making it ideal for the waferscale fabrication of microoptical elements (cf., section 3).

In contrast, OrmoClear® and OrmoCore® are formulations based on the same methacrylate-functionalized hybrid polymer resin where one component used for synthesis is 3-methacryloxypropyl trimethoxy-silane (MEMO), which is a very common alkoxysilane for this type of

synthesis [44]. MEMO supplies one C=C bond per precursor molecule with a moderate photochemical reactivity compared to acrylate-based systems [60]. Notably, the sol–gel reaction for the synthesis of OrmoClear® and OrmoCore® is performed under water-free conditions and a large part of the utilized alkoxysilanes possesses aromatic groups in the organic domain. This results in hybrid polymers with particularly low Si–OH and aliphatic CH contents reducing their absorption at 1310 and 1550 nm and thus making them suitable for the fabrication of single-mode waveguides for data and telecom applications [1, 30].

Multi-nuclei NMR spectroscopy (^{29}Si, ^{1}H) was used to quantitatively follow the evolution of the relative contributions of silicon and carbon atoms during the hydrolysis/(poly)condensation reaction in order to distinguish the different branching of the formed (macro)molecules and to determine the amount of silanol in the resulting pre-polymer material. Figure 3 and Table 1 compare the ^{29}Si-NMR spectra of OrmoComp® and OrmoClear®. For OrmoComp®, the formation of linear siloxane chains and small rings is observed, as evidenced by the presence of the majority of Si groups as D^2 species, while about 28% of the material is present as silanol (D^1). In contrast, for OrmoClear® the inorganic network is much more complicated with a low amount of unreacted alkoxysilanes (D^0, D^1),

linear segments (D^2, T^2) and branching sites (T^3). From this data, the overall inorganic crosslinking can be estimated to be approximately 86 % in case of OrmoComp® and 80 % in case of OrmoClear®. Importantly, while in OrmoComp® free Si–OH groups are detected at the unreacted Si sites, in OrmoClear® and OrmoCore® the alkoxide moieties are still present due to the non-aqueous synthesis environment [44, 46].

The properties which are important to control are the resin's refractive index n_D^{20} (at λ = 589 nm) which was determined with an Abbe refractometer to be 1.493 ± 0.001 for uncured, liquid OrmoComp® and its viscosity which is around 2 Pa s, which is strongly influenced by the synthesis conditions and the work-up procedures. Cured OrmoComp® is highly transparent in the near-UV, visible, and NIR wavelength regimes (cf. Figure 4). Importantly, due to the hybrid nature of the material, transparency is sustained under significant thermal and humidity stress, as observed during aging tests. These prove that the material meets the reliability requirements of wafer-lever optics.

For a controlled fabrication and reliable processing in combined UV lithography/replication and 3D Laser Lithography and for exploration of the full potential of OrmoComp®, a precise control the organic cross-linking reaction is necessary which is achieved by comprehensive understanding of the light-matter interaction processes. Houbertz et al.
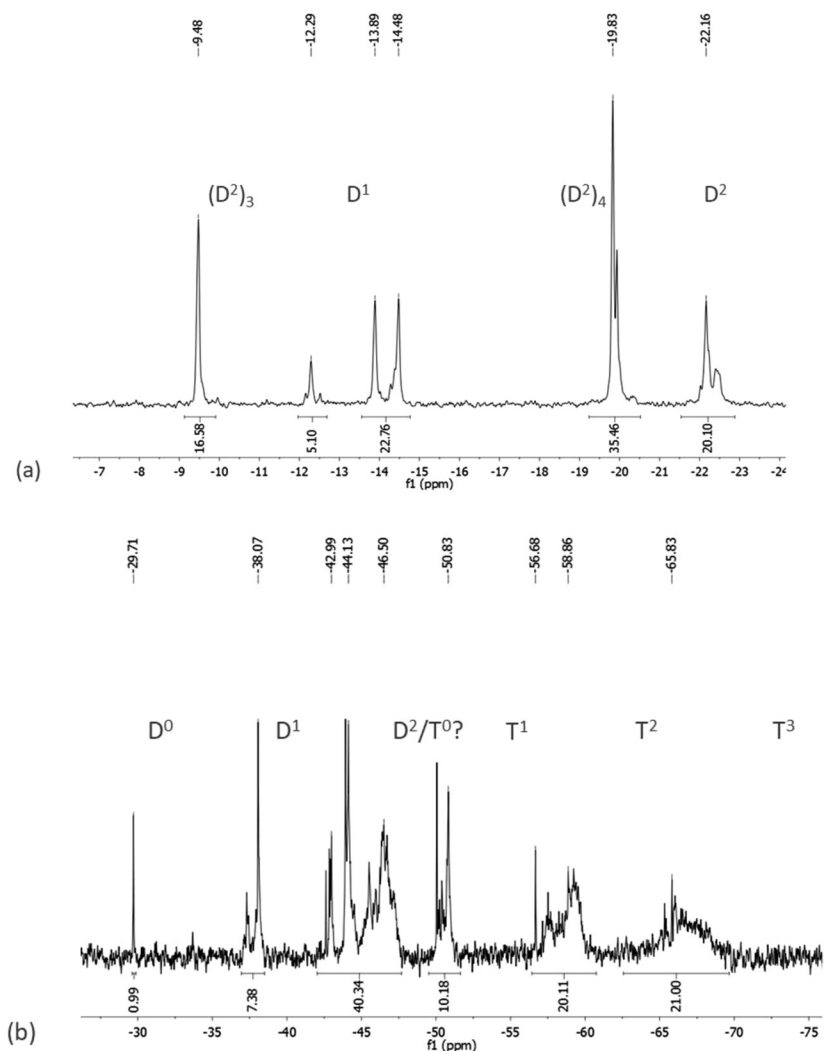
Figure 3: ^{29}Si NMR spectra of (a) OrmoComp® and (b) OrmoClear® with the respective peak assignments, exhibiting the high quality and the high degree of inorganic cross-linking of the hybrid pre-polymer.

Table 1: Siloxane species detected in OrmoComp® and OrmoClear® via ^{29}Si NMR spectroscopy.

OrmoComp®		OrmoClear®	
Species	mol %	Species	mol %
D^1	28	D^0	1
$(D^2)_3$ (3-ring)	17	D^1	8
$(D^2)_4$ (4-ring)	35	D^2/T^0	40
D^2 (large rings, chains)	20	T^1	10
		T^2	20
		T^3	21
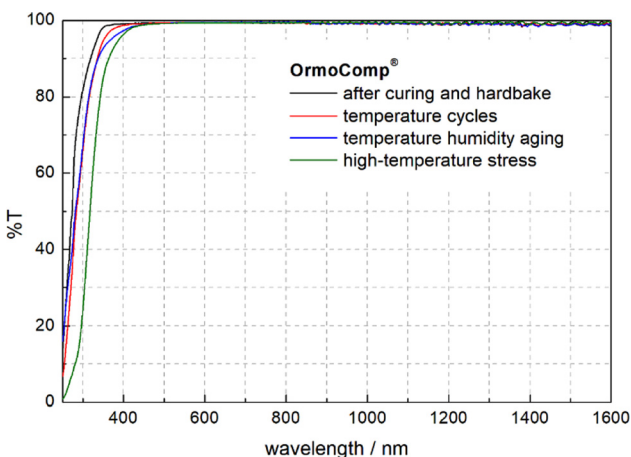
Figure 4: Temperature and climate stability of cured OrmoComp®. The transmission spectra were taken using thin films (d = 20 µm) on quartz glass substrates after UV curing and hardbake (150 °C, 3 h), respectively. Measurements after temperature cycling (−40 °C to 85 °C, 100 cycles), temperature humidity aging (85 °C, 85% RH, 1000 h) as well as high-temperature stress imitating reflow soldering conditions (160 °C–260 °C in 100 min, 2 min hold at 260 °C) are also shown.

[46, 61] have shown using a waveguide material that the optical properties can be precisely controlled via the chemical synthesis conditions and, finally, can be further fine-tuned by controlling the cross-linking condition in the subsequent technological processing in a mask aligner. Therefore, the organic cross-linking of an experimental formulation based on OrmoComp® as inorganic–organic hybrid polymer was investigated by Fourier-Transform Infrared (FTIR) spectroscopy in dependence of the processing conditions. For the spectroscopic measurements, the material was formulated with Irgacure 369 as photoinitiator which is known to initiate the organic cross-linking via a Norrish type I initiation [62]. To follow the impact of photoinitiator concentration on the cross-linking behavior of the material, the concentration of the initiator in the hybrid polymer was varied from 0.025 to 4 wt.% [63]. As substrate material, p-doped Si(100) wafers which were coated with sputter-deposited Au layers were used. The sample preparation for the spectroscopic characterization followed six steps which are depicted in Figure 5(a).

Polymer layers were spin-coated onto the Au/p-Si(100) substrates, and their processing followed a sequence of prebake at 80 °C, UV exposure at defined exposure doses, post-exposure bake at 80 °C, and finally a development step in a solvent such as methylethylketone and p-propanol to remove the inhibition layer formed by oxygen inhibition upon UV exposure. These samples were then characterized by FTIR spectroscopy before and after final thermal annealing at 150 °C for 3 h.

Prior to the FTIR measurements, the stability of the material upon FTIR measurements was tested in dependence of the integration time to evaluate the optimum measurement time. The integration time was varied between 10 and 720 s. As optimum integration time, 120 s were used which is long enough to record low noise data and short enough to not negatively influence the material's behavior in the measurements. Figure 5(b) shows FTIR spectra of the resin, the UV-cured (after step 5, cf., Figure 5(a)) and the same sample after thermal annealing. UV exposure was carried out in a SUSS Mask Aligner (MA6) using broadband flood exposure (peak wavelength 365 nm) at 20 mW/cm^2 for different exposure times, resulting in a variation of exposure doses. For each set of UV and thermal annealing treatment, the same sample was used.

The relevant peaks which are used for the semiquantitative determination [61] of the C=C conversion (or, the degree of organic cross-linking) are assigned in the spectra. In the wave number region of 1720–1750 cm^{-1}, the carbonyl stretching vibrational mode ν(C=O) which is used as internal reference is detected. At 1618 and 1634 cm^{-1}, the vibrational mode ν(C=C) of the acrylate groups is detected which shows a doublet due to the cis–trans isomerism [64]. The initiator's vibrational modes are found at 1599 cm^{-1} for the phenyl deformation mode δ_I(C=C)$_{ar}$, and at 1660 and 1690 cm^{-1} for the initiator's carbonyl mode ν_I(C=O). The peak relation of the cis–trans isomerism in the OrmoComp®-related material was determined to be around 1.46 [64], which corresponds well to the literature value of 1.43 [65]. From Figure 5(b), it becomes obvious that there is no difference in the peak height for the acrylate vibrational mode ν(C=C) for the UV-exposed and the additionally thermally annealed samples. This is a very unique feature of OrmoComp®-related polymers which already reduces the process steps and resources such as energy and time, since principally no final thermal curing has to be carried out.

In order to investigate the influence of the photoinitiator concentration on the organic cross-linking and on the resulting refractive indices, the hybrid polymer was formulated with different amounts of photoinitiator with concentrations ranging from 0.025 to 4 wt.%. The data were taken before and after the development step.

The C=C conversion of the acrylate groups of the OrmoComp® formulation which was determined semi-quantitatively with the C=O (carbonyl) stretching vibrational mode as internal reference is displayed in Figure 6 (a) in dependence of the UV initiator concentration ranging from 0.025 to 4 wt.%. The samples were exposed to UV light at a dose of 600 mJ/cm^2. The measurements were carried out at UV-exposed and additionally thermally annealed layers. Again, no difference in the C=C conversion is observed for the two preparation steps, i.e. OrmoComp® can be processed without thermal annealing. The conversion starts to saturate already for about 0.5 wt.% initiator concentration with a conversion of approximately (67 ± 5) % which does not change by further increasing the amount of initiator. The refractive index was determined using a home-build prism coupler [66] in dependence of the initiator concentration. For reference, the refractive index of the OrmoComp® resin n_D^{20} with initiator was determined with an Abbe refractometer at 587 nm to be 1.495. The slightly higher value as reported before (cf., section 2.2) might be related to the presence of the photoinitiator in the material which has
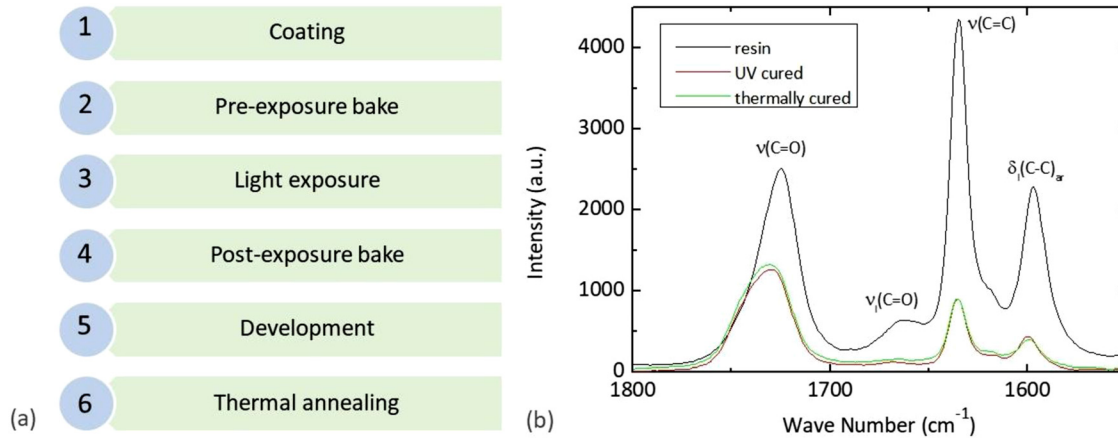
Figure 5: (a) Standard process sequence for the preparation of samples for FTIR characterization. (b) FTIR spectra in dependence of the sample preparation. The data were taken before and after the development step. As reference, the uncured material is also shown.

one phenyl group per molecule which is known to increase the refractive index [67, 68].

The refractive indices of the cured layers were determined at 635 nm and at 1550 nm, respectively. From the measurements at 635 nm it becomes obvious that the values start to saturate as for the C=C conversion at about an initiator concentration of 0.5 wt.%. The maximum refractive index which is achieved with this preparation is 1.522 at 635 nm. Due to the material's dispersion, the refractive index is lower at 1550 nm and amounts to approximately 1.515. The increase in refractive index from the non-cured (i.e., liquid) resin to the fully cured material is about 0.3 (for 635 nm) and about 0.02 (for 1550 nm), respectively, and is relatively independent from the processing conditions in standard UV exposure preparation. The acrylate system reacts different to the UV exposure than the methacrylate system reported by Houbertz et al. [61]. For the methacrylate system, two distinct slopes were observed in the refractive index measurements which can be explained with the formation of differently sized structural units dependent on the initiator concentration. For a small amount of photoinitiator, larger structural units are formed which

result in a lower density compared to a higher amount of initiator, where initiation results in a higher density due to smaller structural units which can be more densely packed. For OrmoComp®, the slope of the curves is less pronounced than for the methacrylate system. This is due to the structural units formed in the material which consists of larger oligomers of 1–2 nm in size and the incorporation of the TMTPA molecules of about 0.6 nm in size, similar to a bimodal structural element distribution where the larger organically modified inorganic-oxidic oligomers and the smaller reactive molecules are cross-linked together. The latter then represent a kind of reactive filler material. A more comprehensive study will be published elsewhere.

3 Application examples

The use of multifunctional inorganic–organic hybrid polymers for waferscale and integrated optics has many advantages compared to other materials, since the number
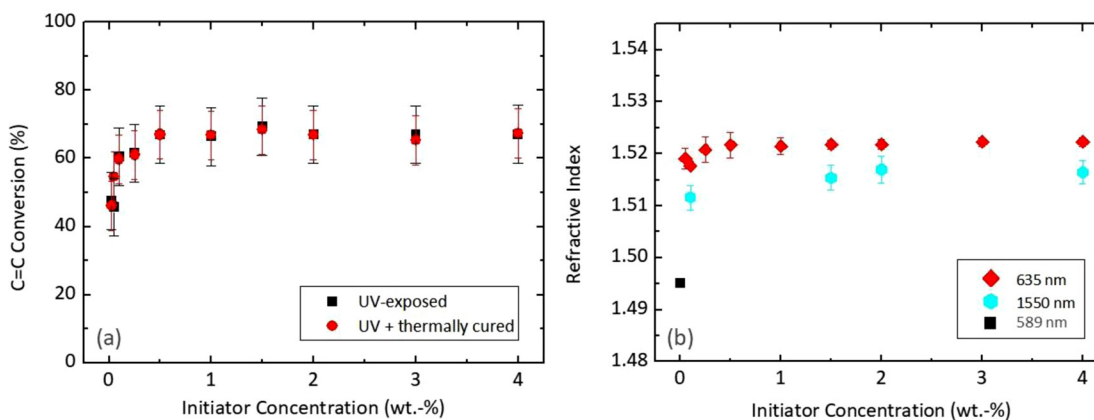
Figure 6: (a) Semi-quantitatively determined C=C conversion of the acrylate groups of OrmoComp® formulated with 0.025–4 wt.% photo-initiator Irgacure 369 for UV-exposed and additionally thermally annealed layers. (b) Refractive index of the cured layers in the VIS (@635 nm) and at 1550 nm determined by prism coupler measurements. The resin's refractive index is also given and was determined with an Abbe refractometer (@589 nm). The samples were UV-cured at an energy dose of 600 mJ/cm^2.

of materials in a package can be significantly reduced. That relates to the fact that the organic functionalities in the material can be chosen such that the material can fulfill many different functions in the package such as planarization, cladding, core, and dielectric layer which was already demonstrated with optical waveguides in PCB [29]. More details will be published elsewhere. The reduction of the number of materials in a packaging concept also reduces the number of interfaces, and thus the probability of failure, since interfaces are critical factors in multilayer processing. Besides, the consumption of resources such as packaging materials, solvents, energy, and process steps can be significantly reduced [69].

The processing of inorganic–organic hybrid polymers such as ORMOCER®s which exhibit negative-tone resist behavior typically consists of two steps: their syntheses resulting in a pre-polymer resin with an organically modified inorganic-oxidic, i.e. an organically modified inorganically cross-linked network and their processing to form an inorganic–organic interpenetrating network by photochemically and/or thermally initiated cross-linking. The processing of the hybrid polymers typically follows a sequence of the material's application onto the substrates and a subsequent curing by UV exposure. For some hybrid materials, a final thermal curing step is recommended, because their cross-linking is not necessarily completed by only UV curing or in order to remove process solvents [45].

Inorganic–organic hybrid polymers such as ORMOCER®s can be patterned from the nanometer up to the centimeter scale, and basically any patterning method available in a micro-structuring lab can be used for structure formation. The most prominent techniques are nano- and micro-patterning via UV lithography, combined UV-assisted replication/lithography, and Laser Direct Writing making use of one- or two-photon processes to fabricate optical elements (see, e.g.

[1–3, 11, 16, 28]). Due to the material's multifunctionality, the number of process steps can be significantly decreased as compared to other material classes [29].

Particularly with respect to waferscale or integrated optical applications, for example for imaging purposes, where the requirements on the optical materials are more challenging than for other applications, inorganic–organic hybrid polymers such as multifunctional ORMOCER®s offer many advantages compared to purely organic materials. For example, the shrinkage of the materials during curing is as low as 5–7% for OrmoComp®, while it can be reduced to below 2% for specific material compositions with high inorganic content. Since there always will be shrinkage resulting from the cross-linking process, residual shrinkage can be compensated in the tool fabrication process by accounting for shrinkage in the optical element's design process which can be very well controlled [70–72].

For waferscale fabrication, double-sided UV-assisted replication in a mask aligner was carried out [28, 30]. As replication tool, a soft elastomer layer on a glass substrate is used. Figure 7(a) shows an overview of a lenslet structure for miniaturized multichannel imaging optics, which is used as mobile miniature microscope. This type of microscopes can be, for example, used for medical purposes in skin scans. Multichannel arrays of microlenses and apertures were fabricated by double-sided replication of OrmoComp® on a 200 mm wafer. The dimensions of the individual arrays which are determined by the size of the imaging chips is $5 \times 9 \, mm^2$ for the used chips; larger arrays are easily possible to fabricate. In Figure 7(b), single-channel imaging optics of individual microlenses with apertures are shown after packaging and dicing. The miniature camera objectives are arranged on $2 \times 2 \, mm^2$ and are used as low resolution optical sensors. More than 5,000 optical elements of this kind can be produced on a 200 mm wafer.
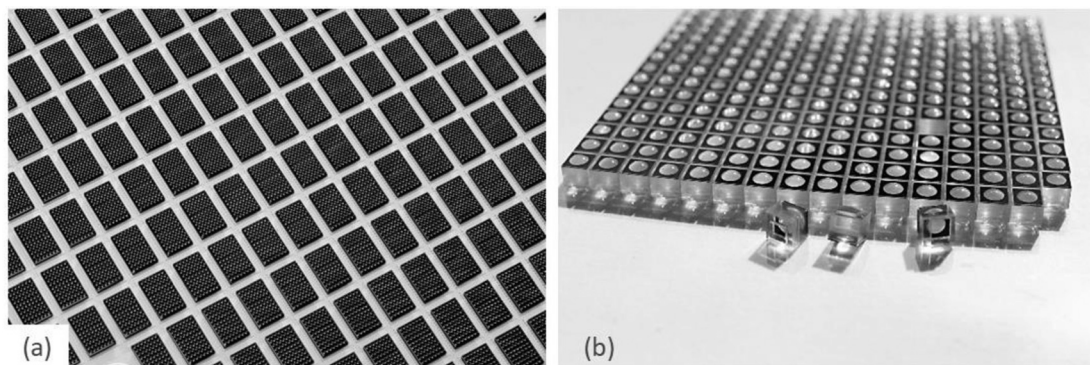
Figure 7: Photographies of lenslet structures (a) for miniaturized multi-channel imaging optics, fabricated by double-sided replication of OrmoComp® on a 200 mm wafer. The dimensions of the individual arrays is $5 \times 9 \, mm^2$, and (b) for single-channel imaging optics after packaging and dicing.

The advantages of the wafer-level technology are especially important in case of a large number of (small) elements or arrays, as well as for the integration of many optical functional layers in devices. Furthermore, its use has advantages in testing and further processing or handling such as, for example pick and place assembly. In combination with the coatability and stability of the hybrid polymer-on-glass optics, solderability is also a major advantage in integration. Depending on the required lateral precision, the transition from currently typical 8 inch wafer size to 12 inch is conceivable.

The additional inorganic network leads to a significantly better mechanical stability. This is not only advantageous for handling and further processing, but also for the stability of all external optical surfaces. Suitable thermally post-cured replica can be coated with plasma-enhanced vapor deposition or sputtering, e.g. as anti-reflection coating. Elements coated this way exhibit better stability properties than many of the purely organic materials. For imaging optical elements, it is sometimes desirable to combine lenses of materials with different dispersion properties to compensate for imaging errors (achromatic doublets). This was demonstrated by well-suited combinations of two different inorganic–organic hybrid polymers such as OrmoComp® and OrmoCore® [73].

Antireflection coated lens arrays of the inorganic–organic hybrid polymer OrmoComp® on glass are now in use for more than 10 years without significant performance degradation (e.g. for micro-optical fiber switches) [74]. OrmoComp® is also used in collimating lenses for vertical emitting laser (VCSEL) arrays [74], and it withstands high intensity light in the NIR range between 800 and 900 nm. These observations on the hybrid material's stability can be confirmed by reliability measurements carried out using edge emitting lasers and microlenses fabricated directly on their facets by Laser Direct Writing using Two-Photon Absorption (TPA) processes, where stability testing is already more than 10,000 h [22].

Another processing technology for direct fabrication of microoptics on waferscale is Laser Direct Writing making use of TPA, where arbitrary 3D structures can be directly additively fabricated from the hybrid pre-polymer OrmoComp® resin [19, 22]. Either individual structures or masters can be fabricated on waferscale, with a tremendous degree of freedom in structure shapes and formats. For that purpose, the material was formulated with photoinitiators typically around 2 wt.% which was found to provide more precise and more stable structuring results than with a lower amount of initiator [75]. From the FTIR measurements (cf., Figure 6), there is no difference in the conversion of the C=C bonds for 1 and 2 wt.% initiator concentration. However, the FTIR data were recorded on samples which were processed

in a mask aligner, i.e. by using one-photon processes, and it was observed that the photochemical cross-linking via TPA leads to denser structures [29, 76]. As typical for all ORMOCER® materials, they are highly transparent in the visible range (cf, Figure 4) which is favorable for the fs laser operating in the VIS at 515–522 nm. Compared to other materials, OrmoComp® exhibits an extraordinary large process window which is useful for process development [77]. Microlenses were fabricated on 4″ and 6″ wafers, respectively, with approximately 24,600 and more than 84,000 individual lenses per wafer [78]. The fabrication is still very time-consuming, however, it is used for the fabrication of replication masters if special freeform or combinations of freeform and other shapes is required. The quality of the structures is strongly dependent on the processing parameters and fabrication modes as well as on the number of writing beams used for fabrication. The quality of the structures when extremely speeding up the process is not yet suited for high-quality optical fabrication, but it shows the way on how to proceed and nicely demonstrates the capability of the 3D technology which has a much better flexibility in structure formation on the same master than any other method.

4 Conclusions

Multifunctional inorganic–organic hybrid polymers offer a cost-efficient and resource-saving way of creating wafer-scale or integrated optics, enabling sustainable processes. The optical properties of these materials were discussed with emphasis on OrmoComp® which was used as optical material for waferscale fabrication. Multi-nuclei NMR and FTIR spectroscopy are presented for different hybrid polymer materials. Due to the material's multifunctionality, most of the processes conventionally used can be saved, for example thermal annealing. At the same time, the material is easy to process and extremely reliable in its performance and lifetime. Application examples of multi- and single-channel imaging devices on waferscale for 200 mm wafers were presented.

Houbertz. B. Hecht (Würzburg University) who had accompanied the works is gratefully acknowledged.

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