

RESEARCH ARTICLE | MAY 15 2014

Lenses for terahertz applications: Development of new materials and production processes **FREE**

N. Kocic; M. Wichmann; T. Hochrein; P. Heidemeyer; K. Kretschmer; I. Radovanovic; A. S. Mondol; M. Koch; M. Bastian

AIP Conf. Proc. 1593, 416–419 (2014)

<https://doi.org/10.1063/1.4873811>



View
Online



Export
Citation

Articles You May Be Interested In

Antimicrobial polymers - The antibacterial effect of photoactivated nano titanium dioxide polymer composites

AIP Conference Proceedings (May 2014)

Color measurement of plastics - From compounding via pelletizing, up to injection molding and extrusion

AIP Conference Proceedings (May 2014)

Robust soft sensor based on an artificial neural network for real-time determination of the melt viscosity of polymers

AIP Conference Proceedings (May 2014)

Lenses for Terahertz Applications: Development of New Materials and Production Processes

N. Kocic^{1*}, M. Wichmann², T. Hochrein¹, P. Heidemeyer¹, K. Kretschmer¹, I. Radovanovic¹, A. S. Mondol², M. Koch², M. Bastian¹

¹SKZ – German Plastics Center, Würzburg, Germany – n.kocic@skz.de; t.hochrein@skz.de; p.heidemeyer@skz.de; k.kretschmer@skz.de; i.radovanovic@skz.de; m.bastian@skz.de

²Department of Physics and Materials Sciences Center, Philipps – Universität Marburg, Germany – matthias.wichmann@physik.uni-marburg.de; abdullah-saif.mondol@hsrw.org; martin.koch@physik.uni-marburg.de

Abstract

Up to now, polymer-based terahertz lenses have been made of pure polymers, such as high-density polyethylene, polytetrafluoroethylene or polypropylene. They are fabricated by injection molding, sintering or mechanical machining. Yet, the refractive indices of the abovementioned materials lie in a range between 1.40 to 1.55. Materials with a higher refractive index would allow for the fabrication of thinner lenses with a smaller curvature, shorter focal lengths and thus improved imaging properties. In this work innovative THz-lenses based on highly filled polymeric compounds, possessing a refractive index significantly higher than that of pure polymers, were developed. Furthermore, a simple and versatile approach for the fabrication of THz-lenses by means of extrusion and subsequent compression molding is presented.

It was shown, that the new lenses exhibit refractive indices around 2.0 and simultaneously a low absorption. Other relevant material properties were satisfying as well. The new lenses show a very good surface quality. Compared to conventional lenses made of pure polymers, the new lenses possess an improved imaging quality and functionality. In addition, substrate lenses filled with highly refracting titanium dioxide could be used as an adequate replacement for their expensive, silicon made counterparts. Furthermore, compression molding proved to be a suitable method to fabricate lenses in a fast, efficient and precise way.

Keywords: highly filled, THz-Lenses, compression molding

INTRODUCTION

The rapid progress of terahertz (THz) systems in the last few years moves this innovative technology out of the focus of a purely scientific interest [1-5] toward commercialisation. There is a large potential for the application of THz systems in the fields of quality control, security, biotechnology, astronomy and communication [6-15]. However, for the commercial use of THz systems passive components such as lenses have to be developed and cost-effectively produced [16-18]. Up to now, lenses used for collimating and focusing the THz beam have been made of pure polytetrafluoroethylene (PTFE), high density polyethylene (HDPE), polypropylene (PP), or other materials from the polyolefin group which possess low THz-absorption and THz-refractive indices between 1.40 and 1.55 [19]. Accordingly, the relatively low refractive indices are the limiting factor regarding focusing power and functionality. A higher refractive index would allow the production of the lenses with a lower curvature. The lower the curvature, smaller can be the lens thickness and thus a better imaging quality would be obtained. Commonly employed manufacturing techniques for the production of collimating and focusing lenses are injection molding or extrusion and subsequent mechanical machining. Due to their lower hardness, polymeric materials cannot be machined with optimum surface quality. This can have a negative influence on the optical properties of the lenses. Substrate lenses are a key component in

photoconductive THz emitters and receivers. They prevent total reflection of the generated THz waves at the boundary layer between the antenna substrate and the free space. In order to enhance the outcoupling efficiency, the substrate lenses have to exhibit a refractive index close to the value of the substrate refractive index which lies around 3.6. Up to now, substrate lenses have been exclusively made of expensive, high-purity silicon. This requires a costly process in which the lenticular shape is grinded from bulk material. Recently was shown, that the refractive index of pure polymers can be enhanced by mixing them with highly refracting additives [20]. Through addition with materials such as TiO₂, ZrO₂, Al₂O₃ and BaSO₄ the refractive index can be adjusted to a great extent. In order to produce very thin lenses with low image defects, a high refractive index and consequently, maximum filler content is desired.

In the scope of this work, the polymer-based THz lenses and their production process were developed. A high refractive index was achieved by adding highly refracting fillers in high concentrations into the polymer matrix. For the first time these highly refracting lenses were produced by compression molding without the need for additional mechanical machining.

EXPERIMENTAL

Raw Materials

The following polymers were used in this study: PP

RJ470 MO (Borealis), HDPE MG9601 (Borealis) as well as a cycloolefinpolymer (COP) Zeonor1060R (Zeon). The main properties of the used polymers are shown in Table 1.

TABLE 1: Main properties of the used polymers

Polymer	MFR (g/10 min)	Density (g/cm ³)
PP	70 ¹	0.905
HDPE	31 ²	0.960
COP	14 ¹	1.010

¹230 °C, 2.16 kg; ²190 °C, 2.16 kg

To increase the refractive index of the pure polymers, titanium dioxide (TiO₂) 2500 (Kronos), aluminium oxide (Al₂O₃) Martoxid MZS1 (Martinswerk) as well as zinc sulphide (ZnS) Sachtolith L (Sachtleben) were used as fillers. Their main properties are listed in Table 2.

TABLE 2: Main properties of the used fillers

Filler	Specific surface area (m ² /g)	Median particle diameter d ₅₀ (µm)	Density (g/cm ³)
Al ₂ O ₃	1.5 – 2.5	1.50 – 1.90	3.9
ZnS	8.0	0.20 – 0.30	4.1
TiO ₂	8.0	0.30 – 0.40	4.2

Sample Preparation

All composites were prepared by melt mixing on a Coperion (model ZSK 26 Mcc) co-rotating twin-screw extruder. Because of the strong abrasive effect of the applied fillers (especially on high contents) wear-protected screw elements and barrels were used.

After compounding, the pellets were air dried at 80 °C for 45 min in a drying chamber and injection molded by using a Battenfeld HM 150 machine. A wear-protected injection molding unit was used. Thereby, the 1A specimens (according to ISO 527) for testing the material properties were produced.

The compositions of all produced samples are shown in Table 3.

TABLE 3: Compositions of produced samples

Sample	Filler	wt.%	vol.%
1	PP/Al ₂ O ₃	75	41.0
2	PP/TiO ₂	75	39.3
3	PP/ZnS	75	39.9
4	HDPE/Al ₂ O ₃	75	42.5
5	HDPE/TiO ₂	75	40.7
6	HDPE/ZnS	70	35.3
7	COP/Al ₂ O ₃	70	37.7
8	COP/TiO ₂	75	41.9

Production of Lenses

The most promising compounds from Table 3 were extruded into cylinders using a single-screw extruder KMBE 1-30-25D from KraussMaffei. The screw and cylinder of the extruder were wear-protected. Afterwards, the extruded cylinders were cut into slices (preforms) and subsequently brought into their final lens by using a press Polystat 200T (Schwabenthan).

Characterization

The tensile properties of the samples were determined according to ISO 527. The Charpy impact strength was determined according to ISO 179-1/1eU. To see the influence of temperature and humidity on the materials, the samples were stored in a climate chamber at 60 °C/50 % r. h. The weight and dimensions of the samples were determined prior and after seven days storage. Furthermore, the samples were immersed in a water bath at 23 °C. Water absorption and dimension change of the samples were determined after seven days according to EN ISO 62. Heat expansion of the samples at the temperature range between 0 °C and 80 °C was determined using a dilatometer according to DIN 53752. The THz properties of the injection molded samples were determined using a standard THz time-domain spectroscopy (TDS) setup. Additionally, characterization of the final lenses with regard to focal spot size was performed using a fiber-coupled THz-TDS setup.

RESULTS AND DISCUSSION

Compounding of Highly Filled Polymers

In this work, filler contents up to 75 wt.% were reached (this corresponds to approx. 40 vol.%). Compounding parameters were selected to obtain a stable process even at high filler contents. Thus, a 12 kg/h throughput at a screw speed of 250 1/min was applied as a basic setting. In general, the compounding process was performed applying a typical temperature profile in the range between 190 °C and 240 °C. The temperature values were adjusted for each material. The melt pressure ranged from 15 bar to 150 bar and was highly dependent on the filler content. Torque and melt temperature were in the range between 18 % and 65 % and between 220 °C and 268 °C, respectively.

It was found that a polymer with a higher MFR value significantly simplifies the compounding of highly filled polymers. In comparison with other used polymers COP has a lower MFR value which led to a partially clogged die plate in the extruder after a while. Thus, melt pressure and melt temperature increased gradually to approx. 150 bar and 265 °C, respectively. Nevertheless, with COP as a polymer matrix the compounds of 75 wt.% TiO₂ and 70 wt.% Al₂O₃ were successfully produced. Among all three used polymers, PP RJ 470 MO proved to be the most suitable for the production of highly filled compounds.

Due to the high filler content, the injection pressure increased occasionally up to 2800 bar. In those cases the injection molding had to be stopped. Additionally, the high amounts of heat conductive mineral fillers caused a rapid solidification of the polymer melt. Sink marks on the surface of the test specimens were also discovered. Hence, the process parameters had to be optimised. In general, high injection speeds (80 cm³/s) as well as a high back pressure (up to 1200 bar) were used. The back pressure was kept up for 25 s.

The mechanical properties of the injection molded test specimen are shown in Table 4.

TABLE 4: Mechanical properties: Young's Module (E_y), tensile strength (σ_t), elongation at break (ϵ_b) and impact strength (σ_i)

Sample	E_y (GPa)	σ_t (MPa)	ϵ_b (%)	σ_i (kJ/m ²)
1	3.5	16.3	0.68	11.8
2	2.7	14.0	0.97	6.0
3	4.1	16.4	0.46	3.6
4	4.2	17.0	0.58	2.8
5	3.0	13.4	0.67	2.1
6	3.3	15.5	0.68	2.1
7	7.6	34.6	0.51	3.9
8	7.3	24.2	0.34	2.6

All samples have a high stiffness due to the high filler contents. COP samples (samples 7 and 8) showed a slightly higher stiffness and strength compared to other samples. Despite to the relatively low toughness (elongation at break and impact strength) due to the very high filler content, all materials meet the requirements for the intended application.

After storing in a conditioning chamber for seven days, all test specimens showed no dimensional change and only marginal weight changes of up to 0.06 wt.%. Water absorption after immersion in a water bath and the determined thermal expansion coefficient are shown in Table 5

TABLE 5: Weight changes after a immersion in a water bath as well as the thermal expansion coefficient (t.e.c.) of the selected samples

Sample	Water absorpt. (wt.%)	t.e.c.x10 ⁵ (K ⁻¹)
1	0.02	8.00
2	0.04	7.76
3	0.46	8.44
4	0.35	8.82
5	0.07	-
6	0.50	10.18
7	0.01	3.46
8	0.01	-

PP/ZnS (sample 3), HDPE/Al₂O₃ (sample 4) and HDPE/ZnS (sample 6) show a slightly higher water absorption in comparison with other materials.

The highly filled compounds show a thermal expansion coefficient which is almost half as high compared to pure polymers [21]. This is one additional reason, why they are more suitable for the production of THz lenses compared to pure polymers.

From the presented results, it follows that all samples are suitable for the production of THz lenses with regard to water absorption as well as mechanical and thermal properties. Accordingly, materials for the lens production were chosen regarding their refractive index (see Table 6) and processibility. Thus, the following material systems were selected as most promising for the production of the lenses: PP/Al₂O₃, PP/TiO₂, as well as PP/ZnS.

Process - Engineering - Solutions Regarding Lens Production

An extrusion tool with an inner diameter of 48 mm and a length of 350 mm was designed and built in order to extrude cylinders, which were used to produce the free-space lenses with a diameter of 50.8 mm (best-form, f-theta and fresnel lenses). A counter-pressure of 30 kg was necessary for a stable extrusion process. The entire principle is shown in Fig. 1.

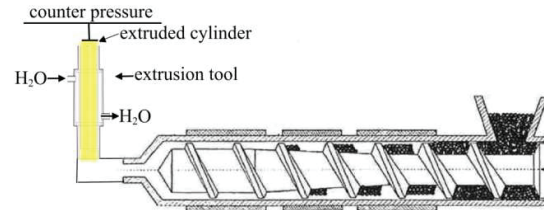


FIGURE 1: Extrusion line for the production of the cylinders of a diameter of 48 mm (schematic drawing)

Additionally, thin strands of 10 mm diameter were required for the production of aspheric and hyper hemispheric substrate lenses and were extruded directly from the extruder die. The extrusion process was performed at a low screw speed between 3 min⁻¹ and 10 min⁻¹. A higher screw speed in the extrusion process means a higher throughput of highly viscous material, which leads to an insufficient cooling of the molten material. A lower throughput also allowed for a better degassing of the melt. A temperature profile in extruder between 160 °C and 210 °C was used. A temperature below 160 °C in the extruder led to poor flow behaviour of the molten material which made the extrusion process impossible. If the temperature was slightly higher than the optimal, the polymer did not solidify in the tool.

It was found that the extrusion process highly depends on the temperature of the upper and lower part of the extrusion tool. A too early solidification of the extrudate in the lower part of the tool led to the sticking of material to the tool wall. Consequently, the torque increased to 80 % so that the extrusion had to be stopped. Accordingly, the temperature in the lower part of the tool had to be increased to ensure a stable extrusion process. For samples filled with Al₂O₃ and TiO₂, the lower part of the tool was tempered at 90 °C and 130 °C, respectively. PP filled with ZnS required a temperature of 200 °C in the lower part of the tool.

Cooling (with water) and solidification took place in the upper tool part (see Fig. 1). The cooling temperature could be adjusted in a range between approx. 20 °C to 80 °C. Low cooling temperature (approx. 20 °C) lead to insufficient material flow. On the other hand, a temperature above approx. 80 °C led to poor cooling. Accordingly, cooling temperatures was kept between 30 °C to 50 °C.

The obtained extrudates were afterwards cut into preforms, which were compression molded to obtain the final lens shape. In order to reduce the tool costs, a changeable molding system was designed. Thus, by

changing the molds in this system, lenses of different geometries were produced easily. Fig. 2 shows the applied press with the molding system.

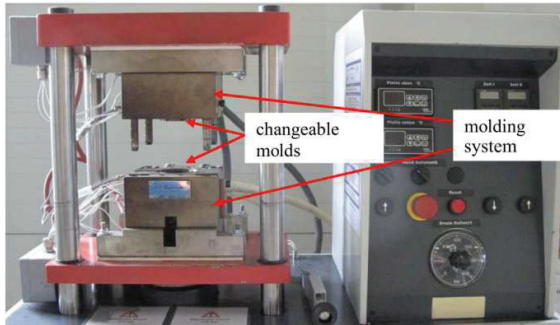


FIGURE 2: Press with the molding system

The lenses were produced as follows: The press was pre-heated up to a certain temperature in the range between 142 °C and 149 °C. This temperature was dependent on the lens type and the material. As soon as the target temperature was reached, the preform was placed into the lower part of the corresponding exchangeable mold. Afterwards, the press was closed and the material was melted for a few minutes under a pressure of 3 bar. Subsequently, the pressure was increased to 100 bar and material was cooled down to room temperature. The surface quality of all produced lenses was very good and thus no additional mechanical machining was needed.

Table 6 gives an overview of all produced lenses, together with their refractive indices.

TABLE 6: Produced lenses and their refractive indices

Lens type	Material	Refractive index
Aspheric	PP+39.1 vol.% TiO ₂	3.02
Hyper hemispheric	PP+40.7 vol.% Al ₂ O ₃	2.09
	PP+39.1 vol.% TiO ₂	3.02
Best-Form	PP+36.1 vol.% Al ₂ O ₃	2.00
	PP+32.4 vol.% ZnS	1.92
f-Theta lens	PP+36.1 vol.% Al ₂ O ₃	2.00
Fresnel lens	PP+39.1 vol.% TiO ₂	3.02
	PP+40.7 vol.% Al ₂ O ₃	2.09

CONCLUSION

In this contribution THz-lenses based on highly filled polymers having a refractive index up to approx. 3.0 were developed and fabricated. The new lenses exceed already existing approaches (e. g. lenses made of pure polymers) in terms of cost efficiency, image quality and functionality. The compression molding proves to be a fast and precise method for the production of THz lenses. With this method, a mass production of simple formed THz lenses becomes possible. This could lead to a significant drop in unit price of the lens. Accordingly, this is an important step toward the realisation of cost-efficient, industrial-suited THz

systems.

ACKNOWLEDGMENTS

We are grateful for the support of the Federal Ministry of Economics and Technology (BMWi) for granting project no. 345 ZN, administered by the German Federation of Industrial Research Associations (AiF).

REFERENCES

1. J. E. Boyd; A. Briskman; C. M. Sayes; D. Mittleman; V. Colvin *The Journal of Physical Chemistry B* 2002, 106, 634.
2. N. Vieweg; B. M. Fischer; M. Reuter; P. Kula; R. Dabrowski; M. Celik; G. Frenking; M. Koch; P. U. Jepsen *Optics Express* 2012, 20, 28249.
3. R. J. Falconer; A. G. Markelz *Journal of Infrared, Millimeter, and Terahertz Waves* 2012, 33, 973.
4. N. Vieweg; M. K. Shakfa; M. Koch *Journal of Infrared, Millimeter, and Terahertz Waves* 2011, 32, 1367.
5. M. Theuer; J. S. Melinger *Journal of Infrared, Millimeter, and Terahertz Waves* 2011, 32, 1267.
6. N. Karpowicz et al. *Appl. Phys. Lett.* 1995, 86, 54105.
7. C. Jördens; F. Rutz; M. Koch in ECNDT Proceedings, Berlin, 2006.
8. C. Jansen. et al. *Applied Optics* 2010, 49, 48.
9. C. Jördens *Optical Engineering* 2008, 47, 037003.
10. N. Krumbholz; T. Hochrein; N. Vieweg; T. Hasek; K. Kretschmer; M. Bastian; M. Mikulics; M. Koch *Polymer Testing* 1995, 28, 30.
11. T. Klein-Ostmann; T. Nagatsuma *Journal of Infrared, Millimeter, and Terahertz Waves* 2011, 32, 143.
12. J. F. O'Hara; W. Withayachumnankul; I. Al-Naib *Journal of Infrared, Millimeter, and Terahertz Waves* 2012, 33, 245.
13. V. Astley; K. S. Reichel; J. Jones; R. Mendis; D. M. Mittleman *Applied Physics Letters* 2012, 100, 231108.
14. Siegel, P; H *IEEE Transactions on Microwave Theory and Techniques* 2002, 50, 910.
15. Tonouchi, M *Nature Photonics*, 2007, 1, 97.
16. B. Scherger; C. Jördens; M. Koch *Optics Express* 2011, 19, 4528.
17. B. Scherger; M. Scheller; C. Jansen; M. Koch; K. Wiesauer *Applied Optics* 2011 50, 2256.
18. M. Wichmann; B. Scherger; S. Schumann; S. Lippert; M. Scheller; S. F. Busch; C. Jansen; M. Koch *Optics Express* 2011 19, 25151.
19. Y. Jin; G. Kim *Journal of the Korean Physical Society* 2006, 49, 513.
20. S. Wietzke; C. Jansen; F. Rutz; D. Mittleman; M. Koch *Polymer Testing* 2007, 26, 614.
21. G. W. Ehrenstein, *Polymer-Werkstoffe: Struktur-Eigenschaften-Anwendung. 2. völlig überarbeitete Auflage*, Carl Hanser Verlag, München, 1999.