DEVELOPMENT OF FLEXIBLE MATERIALS FOR PHOTONIC OPTICAL SKINS

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Abstract – In the present work, we report on the application of flexible polymethacrylates for optical applications. A variety of copolymers with varying glass transition temperature (T_g) was developed and characterised using classical structural and thermal analysis techniques, including ^{1}H-NMR spectroscopy, DSC- and TGA- analysis. The results indicate the potential of the materials developed for flexibilising the current generation of optical materials.

Introduction

During the last decade, the development of smart flexible skins has attracted large attention for a variety of applications such as tactile sensors. [1]-[4] In the present work, we report on the development of flexible polymethacrylate based copolymers which will be applied for the embedding of optical fibres, waveguides and data processing units. For the waveguide approach, the polymers developed will be combined with commercially available formulations.

Experimental

Materials

All monomers, methyl methacrylate (Aldrich, 99%), butyl methacrylate (Acros, 99%) and 2-ethylhexyl methacrylate (Fluka, +98%) were distilled for purification before use. Toluene was dried over calcium hydride before use. As initiator, 2,2'-azobis(2-methylpropionitrile) (Acros, 98%), was used without any further purification.
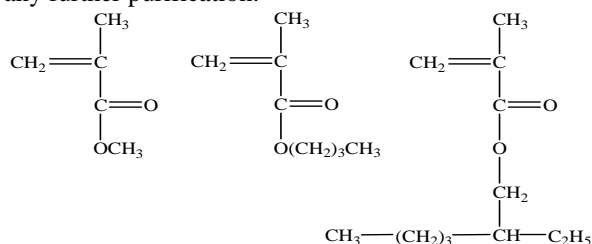

Figure 1: Overview of monomers: methyl-methacrylate (MMA, left), butyl methacrylate (BuMA, middle) and 2-ethylhexyl methacrylate (EHMA, right)

Methods

Structural characterisation of the (co-)polymers was performed by means of ^{1}H-NMR in CDCl$_3$. Spectra were recorded on a Brüker 300 MHz spectrometer. Thermal properties of the materials were determined by means of DSC (differential scanning calorimetry) and TGA (thermogravimetric analysis) measurements on TA Instruments equipment DSC 2920 Modulated DSC and Hi-Res TGA 2950 Thermogravimetric Analyzer respectively.

General procedure for polymerization of methyl methacrylate

Methyl methacrylate (3 ml, 28.05 mmol) was dissolved in toluene (12 ml) in a 2 neck flask. The solution was degassed 3 times and placed under nitrogen atmosphere. The desired amount of radical initiator, 2,2'-azobis(2-methylpropionitrile), was then added as a 10 mg/ml solution in toluene. Before reaction at 75°C, the mixture was degassed once more and the set-up was placed under nitrogen atmosphere. After 24h, the final polymer was isolated as a white powder by precipitation of the reaction mixture in a 10-fold excess ice cooled pentane. The polymer was filtered and dried. Further purification was performed by means of dialysis and redissolution and precipitating.

General procedure for production of MMA containing copolymers

The procedure is similar as described above for PMMA. Instead of 28.05 mmol methyl methacrylate, 28.05 mmol of monomer mixture (e.g. 22.44 mmol MMA and 5.61 mmol BuMA for a copolymer with molar ratio MMA/BuMA 80/20) was dissolved in 12 ml toluene. Further steps were identical except for the precipitation. Depending on the copolymer composition, pentane or methanol was used as non-solvent to precipitate the final product.

Results and Discussion

In order to be sure that the obtained copolymers had the desired composition, ^{1}H-NMR spectra of all the products were recorded. By means of integration of the peaks, we were also able to determine the experimental composition of the different materials. Table 1 shows that there was a good correlation between the theoretical and experimental composition of the developed copolymers.

Table 1: Theoretical & experimental composition of the copolymers developed

Code	Theoretical composition (mol%)	Experimental composition (mol%)
TVG1	MMA/BuMA 80/20	MMA/BuMA 75/25
TVG2	MMA/BuMA 50/50	MMA/BuMA 42/48
TVG3	MMA/BuMA 20/80	MMA/BuMA 24/76
TVG4	MMA/EHMA 80/20	MMA/EHMA 79/21
TVG5	MMA/EHMA 50/50	MMA/EHMA 51/49
TVG6	MMA/EHMA 20/80	MMA/EHMA 21/79

One field of application of the different materials is the flexibilisation of Truemode[TM] waveguides. The aim is to create more flexible systems by means of admixing copolymers. A first indication about the flexibility of the copolymers developed can be given by the glass transition temperature (T_g) of the different materials, since the T_g of a material decreases with increasing flexibility. Table 2 shows the results of the DSC measurements performed

Table 2: DSC- & TGA-analysis of the copolymers developed

Code	T_g (°C)	T_{decomp} (°C)
TVG1	95.85	---
TVG2	65.62	---
TVG3	51.13	---
TVG4	51.37	221.84
TVG5	29.40	229.95
TVG6	- 4.19	228.93

It can be observed that the glass transition temperature can be substantially lowered by partial replacement of MMA by BuMA. The effect is even more pronounced when EHMA is used.

To test the possible influence of the copolymers developed on the flexibility of a processed Truemode[TM] formulation, different mixtures with a varying Truemode[TM]/copolymer ratio were processed. Qualitative analysis of the results obtained indeed confirmed an increased flexibility upon copolymer admixing.

Since high temperatures are used in the waveguide processing, the thermal stability of the materials was tested using TGA analysis. The temperatures at which 95% of the material was still intact, T_{decomp}, are depicted in table 2 for the EHMA-containing copolymers. It can be derived from the data that the copolymers developed are compatible with the Truemode[TM] processing scheme, where maximum temperatures of 220-230°C are applied.

Another track where the copolymers will be applied, is as material for the embedding of optical fibres. To get an idea about the embedding capacities and a possible influence on optical fibre properties, an UV transparent glass mould was fabricated by ultrasonic milling. Fibre alignment tools were produced by micromilling in stainless steel. The mould is depicted in Figure 2.
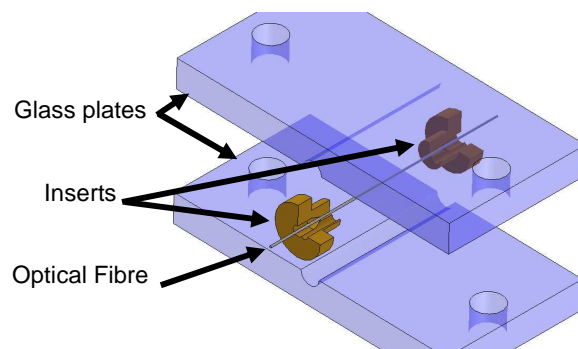

Figure 2: Glass mould

At present, the mould developed is being applied for fibre embedding purposes. The initial results will be presented during the meeting.

Conclusions

In the present work, we have developed series of methacrylate based copolymers to be applied in the field of optical applications. For the different applications envisaged, flexible materials are a strong requirement. Therefore, the building blocks of the copolymers developed were finetuned in order to develop low T_g-materials. At present the materials are screened for the embedding of optical fibres, waveguides and data processing units.

Acknowledgements

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