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# Study of Optical and Thermo-Optical Properties of a Hybrid Photopolymer Material Based on Thiol-Siloxane and Tetraacrylate Oligomer

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**Abstract**—Optical (refractive index and absorption coefficient) and thermo-optical (linear thermal expansion and thermo-optic coefficients) characteristics of a new hybrid organic-inorganic photopolymer material “Hybrimer-TATS” based on thiol-siloxane and tetraacrylate oligomers are studied. Variation of the ratio of initial components makes it possible to change the thermo-optic coefficient from  $-0.7 \cdot 10^{-4}$  to  $0.66 \cdot 10^{-4} \text{ K}^{-1}$ , which offers prospects for synthesizing athermal optical components and optical elements with a high sensitivity to temperature variation for thermal sensors.

**Keywords:** hybrid photopolymer, thiol-siloxane and tetraacrylate oligomers, refractive index, thermo-optic coefficient, thermal expansion coefficient, athermalization.

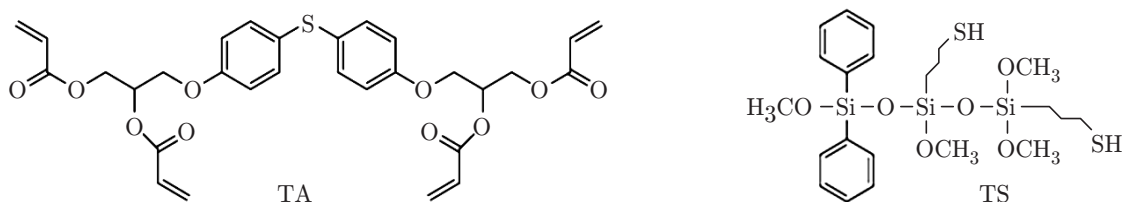
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## INTRODUCTION

Hybrid organic-inorganic photopolymer compositions have been intensely studied for the last two decades. The properties of these materials depend on the composition and can be smoothly and deliberately varied by changing the ratio of the components in the initial photopolymer composition. The most well-known and commercially available photopolymer materials are Ormocer (Organically Modified Ceramics) and SU-8. Ormocer is a photocurable hybrid material, which includes several variants of the composition for different technologies of fabrication of microoptical components (OrmoComp, InkOrmo, OrmoClear, OrmoCore, OrmoClad, and OrmoStamp) [1].

Thermo-optical and thermo-mechanical properties of photopolymer materials are important for creating elements of integrated and diffraction optics: optical switches, variable optical attenuators, optical connectors/splitters, and arrayed waveguide gratings.

On the one hand, a large thermo-optic coefficient (TOC) adversely affects the properties of arrayed waveguide gratings because the emission wavelength is shifted as the refractive index of waveguides changes [2]; on the other hand, it allows creating optical sensors based on fiber or planar Bragg gratings [3]. Athermal



**Fig. 1.** Structures of tetraacrylate and thiol-siloxane monomers-oligomers in the composition of the hybrid photopolymer material Hybrimer-TATS.

waveguide devices whose optical characteristics are independent of temperature variations are necessary for creating multiplexers for splitting of the light flow in wavelength order [4].

Thus, it is important to know thermo-optical characteristics of hybrid photopolymer materials for the development of various functional elements of the optical system.

Shelkovnikov et al. [5] synthesized and studied the thermo-mechanical properties of “cross-linked” solid films of a hybrid photopolymer material Hybrimer-TATS obtained by means of photopolymerization of a composition consisting of tetraacrylate (TA) and thiol-siloxane (TS) oligomers with different ratios of these components. The structures of such oligomers are shown in Fig. 1.

It was found that photopolymerized Hybrimer-TATS films exhibit a high thermal stability (up to 300 °C) in both inert and oxidizing media, while the thermo-mechanical properties are conditioned by the ratio of TA and TS in the initial composition. Therefore, the thermo-optical properties of the material can be expected to be different depending on the ratio of the TA and TS oligomers.

The goal of the present work is to study the influence of the ratio of the initial oligomers on the thermo-optical characteristics of the photocured hybrid photopolymer material Hybrimer-TATS.

## EXPERIMENTAL RESULTS AND DISCUSSION

### *Synthesis of Hybrid Photopolymer Compositions and Experimental Samples of Hybrimer-TATS*

To perform the measurements, we synthesized a number of photopolymer compositions, which were solutions consisting of chloroform, photoinitiator, and a mixture of TA and TS oligomers with different ratios of these components. Photopolymerization was initiated by 2,4-diethyl-9-oxo-10-(4-heptyloxyphenyl)-9H-thioxanthenium hexafluorophosphate in the form of an additive (5 wt.%) to the solution of TA oligomers in chloroform. Addition of the photopolymerization initiator leads to enhancement of absorption in the range of 370–430 nm.

To measure the refractive index, the composition was deposited onto a glass substrate, dried until complete volatilization of the solvent, and exposed to UV radiation in an ELC-500 UV chamber (produced by the Electro-light Corporation) for 10 min until a solid film of a cross-linked polymer was obtained. As a result, the samples consisted of layers of a hybrid photopolymer material 100–200  $\mu\text{m}$  thick.

The samples for measurements of the thermo-optical properties were made in the form of a “sandwich” where the examined material is enclosed between two glass plates. The sandwiches of photocured Hybrimer-TATS were fabricated in the following manner: 1 ml of the 30% solution of the TA and TS oligomers in chloroform were dried until complete volatilization of the solvent at a temperature of 50 °C during 30 min, after which 50  $\mu\text{l}$  of the viscous photopolymer composition were poured between 10  $\times$  10 mm plates made of borosilicate glass. The thickness of the examined material was determined by the thickness of plane-parallel inserts placed between the glass plates. To prevent cracking in the thick layer, dark pre-polymerization was performed during 15 days, followed by 10-min photopolymerization in the ELC-500 UV chamber. No thermal treatment of the resultant sample was required.

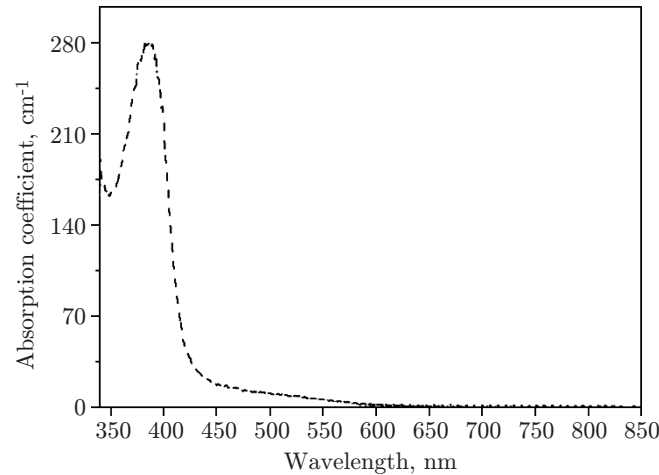
### *Measurement of the Refractive Index*

The refractive index of Hybrimer-TATS before and after photopolymerization was measured by an IRF-454BM refractometer; samples with different mole fractions of the TA and TS oligomers were tested.

It is seen from Table 1 that differences in the measured refractive indices of the Hybrimer-TATS samples due to variations of the ratio of the TA and TS oligomers and also between the states before and after photopolymerization are observed in the third decimal place.

**Table 1**

TA : TS ratio	Refractive index $n$	
	before photopolymerization	after photopolymerization
2 : 1	1.5205	1.5255
1 : 1	1.5211	1.5251
1 : 2	1.5225	1.5248

**Fig. 2.** Absorption spectrum of the Hybrimer-TATS film 170  $\mu\text{m}$  thick.

The growth of the refractive index in the course of the polymerization reaction is apparently related to the increase in material density. During polymerization of molecules, the double bonds of the TA oligomer are opened, forming the cross-linked polymer network. Such changes lead to an increase in material density.

In terms of creation of microstructured optical components, the refractive index of the photocured material Hybrimer-TATS ( $n = 1.525$ ) is close to the refractive index of widely used Crown optical glasses ( $n = 1.517$ ). This offers a possibility of forming a microstructured optical component in a thin polymer layer on a glass substrate. Moreover, the interface between the glass and the Hybrimer-TATS layer does not reflect light and does not produce spurious flares.

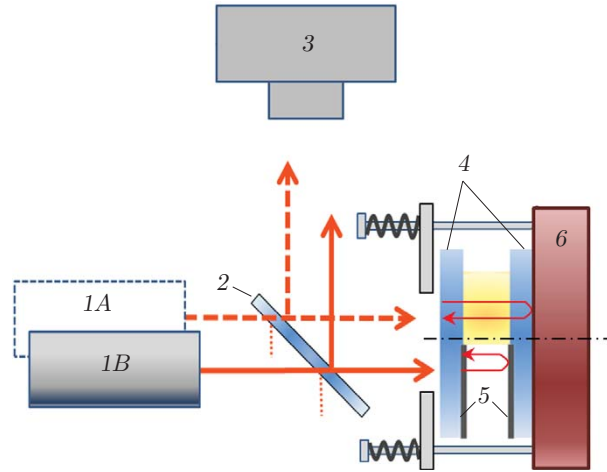
#### *Measurement of the Absorption Coefficient of Hybrimer-TATS*

The absorption coefficient is an important parameter in forming waveguide structures and thick microoptical elements based on the photopolymer material Hybrimer-TATS. To measure the absorption spectrum, we covered a glass substrate by a Hybrimer-TATS layer 170  $\mu\text{m}$  thick with the TA : TS ratio equal to 2 : 1. The absorption spectrum of the photopolymer was determined by using the Beer–Lambert law with allowance for absorption by the substrate.

It follows from Fig. 2 that Hybrimer-TATS films ensure intense absorption in the UV range 350  $\div$  400 nm, which is related to absorption of products of initiator photodecomposition. These films also exhibit weak absorption in the range 450  $\div$  650 nm due to formation of a complex with charge transfer between the photoinitiator and the TA monomer. Transparency of this material in the visible spectral range starts from 600 nm, which makes it suitable for creating components of diffraction, microoptical, and integrated optical elements operating in the red and near-infrared spectral ranges.

#### *Study of Thermo-Optical Properties*

To measure the thermo-optic coefficient and the coefficient of linear thermal expansion of the examined material, we use the interference method [6, 7] and a Fabry–Perot multibeam interferometer. The material sample is placed between the interferometer mirrors. When the sample is heated in the thermostat, its refractive index and thickness change, resulting in changes in the phase of the interfering light beams passing through the sample and, hence, in shifting of the interference pattern. The experimental setup is schematically shown in Fig. 3.



**Fig. 3.** Experimental setup: (1A, 1B) positions of the He-Ne laser in the measurement configurations A and B, (2) beam splitter, (3) USB camera, (4) sandwich with the examined material, (5) semitransparent mirrors, and (6) thermostat.

The source of radiation is a He-Ne laser 1 with a wavelength of 633 nm. In the TOC measurement mode (configuration A), the beam of the laser located in the position 1A passes through the beam splitter 2 and impinges onto the Fabry–Perot resonator 4 shaped as a sandwich with the examined material between two glass plates. The light beams reflected from the external surfaces of the sandwich are further reflected from the beam splitter and form an interference pattern recorded by a USB camera 3 connected to a computer. In the mode of measurement of the thermal expansion coefficient (TEC) (configuration B), the beam of the laser located in the position 1B passes through the beam splitter and impinges onto sandwich regions where the glass plates do not contact the examined material Hybrimer-TATS. The internal surfaces of these regions are semitransparent mirrors 5 (deposited chromium film with 40% transmission). They are necessary to increase the contrast of the corresponding interference pattern.

The sandwich temperature is controlled by a thermostat 6 with a step of 0.1 °C in the temperature interval from 25 to 70 °C. The sample holder is a heat-conducting ring, which is fixed on the thermostat flange with the use of weakly springy fixtures. This method of sample fixation does not prevent its thermal expansion. When the sandwich is heated, the interference fringes start to move. When a prescribed temperature is reached, the heater circuit is interrupted. The shift of the interference fringes with respect to their positions at the beginning of heating is determined. The shift of the interference fringes is also measured in the course of sample cooling to check the reproducibility of results.

The relative error of measurements of the temperature and shift of the interference fringes varies from 2.2 to 6.6% at temperatures above 60 °C and approximately from 1 to 5% in the case of a strongly tapered sample. This corresponds to the relative measurement error of the optical path difference from 3.2 to 11.6%. The data necessary for error estimation were accumulated in multiple measurements of the characteristics of a reference sample.

The method of calculating the TOC and TEC in the present work differs from that described in [6, 7]: the Fabry–Perot resonator is shaped as a sandwich rather than a solid block. The optical path difference ( $\Delta L$ ) of the beams incident onto the sandwich and reflected from its external surfaces can be calculated as

$$\Delta L = 2L_1n_1 + 4L_2n_2 + \lambda/2, \quad (1)$$

where  $L_1$ ,  $L_2$  and  $n_1$ ,  $n_2$  and the thicknesses and refractive indices of the examined material and glass plates in the sandwich, respectively;  $\lambda/2$  is the halved wavelength added to the path difference owing to reflection of the beams from a denser medium.

Differentiating Eq. (1) with respect to temperature, we obtain the coefficient of the thermally induced optical path difference  $d\Delta L/dT$  for the sample in the measurement configuration A:

$$\frac{d\Delta L_b}{dT} = 2L_1 \left( n_1\alpha_1 + \frac{dn_1}{dT} \right) + \phi, \quad (2)$$

**Table 2**

Material		$n$ ( $\lambda \approx 633$ nm)	TOC ( $dn/dT, 10^{-4} \text{ K}^{-1}$ )	TEC ( $\alpha, 10^{-4} \text{ K}^{-1}$ )
Hybrimer-TATS	1 : 4	1.525	0.66	0.7
	1 : 2		0.43	0.71
	1 : 1		-0.06	0.61
	2 : 1		-0.43	0.56
	14 : 1		-0.7	0.50
SU-8 [8]		1.59	-1.1	0.52
OrmoComp/InkOrmo [9]/[1]		1.52	-1.3/-2	0.6
OrmoClear/OrmoCore/OrmoClad [1]		1.537 ~ 1.555	-2.1/-2.2/-2.7	1 ~ 1.3
OrmoStamp [1]		1.516	-1.5	1.05
PMMA [10, 2]/measured		1.49	-0.80 ~ -1.3/-1.3	0.9 ~ 2.2/0.9
Polycarbonate [10]		1.59	-1.07	0.7
Borosilicate glass [11]		1.517	0.025	0.033

where  $\phi = 4L_2(n_2\alpha_2 + dn_2/dT)$  is the coefficient of the thermally induced optical path difference in the glass plates,  $\alpha_{1,2} = (1/L_{1,2})(dL_{1,2}/dT)$  is the linear thermal expansion coefficient, and  $dn_{1,2}/dT$  is the thermo-optic coefficient of the examined material and glass. Using the values of TOC, TEC, and refractive index known for borosilicate glass, we obtain  $\phi = L_2(0.6 \cdot 10^{-5}) \text{ K}^{-1}$ .

In the measurement configuration  $B$ , where the examined material is absent in the beam path, Eq. (2) transforms to

$$\frac{d\Delta L_a}{dT} = 2L_1n_1\alpha_1. \quad (3)$$

In this case, the beams reflected by the semitransparent mirrors applied onto the internal surfaces of the glass plates interfere with each other.

The beams reflected from the sample (Fabry–Perot resonator) form an interference pattern consisting of fringes with an identical slope, which are detected by the camera. The shift by one interference fringe corresponds to a change in the optical path difference inside the resonator by one half of the wavelength of the probing radiation. The coefficient of the thermally induced optical path difference of the beams in the irradiated region of the sample is determined as

$$\frac{\delta\Delta L_{a,b}}{\delta T} = \frac{\lambda}{2} \frac{\delta m_{a,b}}{\delta T}, \quad (4)$$

where  $\lambda$  is the probing radiation wavelength,  $\delta m_{a,b}$  is the number of interference fringes shifted during the period needed for the material temperature to change by  $\delta T$  in the measurement configuration  $A$  or  $B$ .

Equation (3) for each configuration can be substituted into Eqs. (1) and (2). Transforming Eq. (3), we can express the TEC via the shift of the fringes of the interference pattern as

$$\alpha_1 = \frac{1}{2n_1L_1} \frac{\lambda}{2} \frac{\delta m_a}{\delta T}. \quad (5)$$

The TOC of the examined material is defined by the difference between Eqs. (2) and (3) with allowance for Eq. (4):

$$\frac{dn_1}{dT} = \frac{1}{2L_1} \left( \frac{\lambda}{2} \frac{\delta(m_b - m_a)}{\delta T} - \phi \right). \quad (6)$$

Using Eqs. (5) and (6), we obtained the experimental values of TOC and TEC, which are listed in Table 2 for the photocured material Hybrimer-TATS with different mole fractions of the TA and TS oligomers. Available values of these coefficients for polymers, photopolymers, and glasses are also given in Table 2.

As it follows from Table 2, the TOC and TEC can be deliberately changed by varying the composition and tend to increase with increasing fraction of the TS oligomers. As a whole, the measured coefficients are close to the TOC and TEC values for SU-8, OrmoComp, and polycarbonate. The TOC and TEC values for borosilicate glass are significantly smaller (by an order of magnitude).

The TEC has the value of  $0.6 \cdot 10^{-4} \text{ K}^{-1}$  for OrmoComp and InkOrmo and lies in the interval  $(1 \div 1.3) \cdot 10^{-4} \text{ K}^{-1}$  for the remaining materials. The TOC value varies from  $1.5 \cdot 10^{-4} \text{ K}^{-1}$  for OrmoStamp

to  $2.7 \cdot 10^{-4} \text{ K}^{-1}$  for OrmoClad. These parameters illustrate optimization of the material composition for different technologies. Commercially available Ormocer polymers in the form of a single-species composition immediately suitable for photopolymerization does not allow the user to develop a material with optimal properties for a particular type of microoptical elements.

It is of interest to note that the TOC of the Hybrimer-TATS material acquires a negative value as the fraction of the TA component is increased. This feature can be used to create athermal subwavelength resonant waveguide gratings [9]. Moreover, this fact offers a possibility of compensating for the increase in the beam path difference in the refractive material based on Hybrimer-TATS induced by thermal expansion at the expense of reduction of the refractive index.

Let us consider this effect as applied to a refractive microlens made completely of the Hybrimer-TATS material and located in an air medium. Such microlenses are used for optical interconnects [12]. The general expression for the increment  $\Delta f$  of the focal distance  $f$  of the lens as a function of the change in temperature  $\Delta T$  is

$$\Delta f = f \Delta T x_f, \quad (7)$$

where  $x_f$  is the coefficient of opto-thermal expansion. For a refractive lens in an air medium, this coefficient can be calculated by the formula [13]

$$x_f = \alpha - \frac{1}{n - n_{\text{air}}} \left( \frac{dn}{dT} - n \frac{dn_{\text{air}}}{dT} \right),$$

where  $n$  and  $\alpha$  are the refractive index and the linear expansion coefficient of the lens material. It is seen from this expression that lens athermalization occurs (i.e., the focal distance of the lens becomes independent of temperature) under the condition

$$\alpha = \frac{1}{n - n_{\text{air}}} \left( \frac{dn}{dT} - n \frac{dn_{\text{air}}}{dT} \right). \quad (8)$$

The thermo-optic coefficients of all polymers is greater than that of air approximately by two orders of magnitude. For a polymer with a refractive index about  $n = 1.5$  to satisfy Eq. (8), the absolute value of its TOC should be positive and twice smaller than the TEC of this material. It is seen that the TOC is negative for available polymers, including hybrid polymers, and a lens made of such materials cannot exhibit an athermal behavior. At the same time, it follows from Eq. (8) and Table 2 that, by varying the ratio of the TA and TS components of the examined Hybrimer-TATS material between 1:1 and 1:2, it is possible to find a TA:TS ratio with the thermo-optic coefficient in the interval  $(0.31 \div 0.35) \cdot 10^{-4} \text{ K}^{-1}$ , which ensures athermalization of the refractive lens made of this material for the values of  $\alpha$  in the interval  $(0.61 \div 0.71) \cdot 10^{-4} \text{ K}^{-1}$ .

Such an athermal refractive lens offers an advantage over an athermal hybrid diffractive-refractive lens owing to smaller energy losses and a possibility of using an antireflection coating. It is known that application of an antireflection coating onto the surface of the diffractive component leads to smoothing of the relief of this component and to significant reduction of the diffraction efficiency.

A promising area of applicability of the Hybrimer-TATS material is the development of integrated optical waveguide elements. Chekhlova et al. [14] derived an expression for the central frequency  $\lambda_c$  of spectral multiplexers/demultiplexers as a function of temperature

$$\frac{d\lambda_c}{dT} = \lambda_c \left( \frac{1}{n_{\text{eff}}} \frac{dn_{\text{eff}}}{dT} + \alpha_{\text{sub}} \right), \quad (9)$$

where  $n_{\text{eff}}$  is the effective refractive index of the waveguide material and  $\alpha_{\text{sub}}$  is the linear expansion coefficient of the substrate. The effect of athermalization arises when the expression in the brackets vanishes. For a waveguide made of the Hybrimer-TATS material on a silicon substrate with  $\alpha_{\text{sub}} = 2.6 \cdot 10^{-6} \text{ K}^{-1}$ , athermalization can be ensured by carefully choosing the components with the TA:TS ratio close to 1:1 (see Table 2).

There is another challenge in addition to the problem of athermalization of optical components and optical schemes: creation of components with a high sensitivity to changes in temperature for optical sensors suitable for operation in those regions where electric thermometers cannot be used because of explosion hazard or interference of electric fields. One of the simplest temperature-sensitive components of an optical

sensor is the Fabry–Perot resonator [15]. It follows from Eq. (3) that the coefficient of the thermally induced optical path difference increases if  $\alpha_1$  and  $dn_1/dT$  have the same sign. It is seen from Table 2 that the photopolymerized material Hybrimer-TATS with the TA : TS ratio equal to 1 : 4 in the initial composition yields the value of  $n_1\alpha_1 + dn_1/dT$  equal to 1.73, which is inferior only to some types of PMMA. Owing to its chemical and mechanical permanence, Hybrimer-TATS is a promising structural material for optical sensors of temperature.

## CONCLUSIONS

The refractive index, thermo-optic coefficient, and thermal expansion coefficient of the photocured Hybrimer-TATS material based on tetraacrylate and thiol-siloxane oligomers with different ratios of these components in the initial composition are measured. It is demonstrated that, by varying the ratio of the initial components, it is possible to change the thermo-optic coefficient of Hybrimer-TATS from  $-0.7 \cdot 10^{-4}$  to  $0.66 \cdot 10^{-4} \text{ K}^{-1}$  and to change the thermal expansion coefficient from  $0.5 \cdot 10^{-4}$  to  $0.7 \cdot 10^{-4} \text{ K}^{-1}$ . Variations of these characteristics of the Hybrimer-TATS material may be beneficial for using this material for creating both optical sensors of temperature and athermal microoptical and waveguide components, e.g., refractive microlenses for optical interconnects and spectral multiplexers/demultiplexers.

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