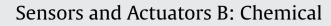
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## Humidity and temperature induced changes in the diffraction efficiency and the Bragg angle of slanted photopolymer-based holographic gratings

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#### ABSTRACT

This work explores the humidity and temperature response of volume phase slanted gratings recorded in photopolymers with varied chemical composition. Acrylamide and diacetone acrylamide were used as monomers and triethanolamine and *N*-phenylglycine were used as photoinitiators. The study demonstrates that the response of photopolymer-based holographic gratings to relative humidity (RH) and temperature (T) can be tuned by alteration of the photopolymer composition.

Humidity and temperature response of the holograms has been characterized by recording Bragg selectivity curves of transmission gratings and by monitoring the position of the maximum intensity in the spectral response of reflection gratings. Investigation of the humidity response in the range of 20–90% RH reveals that photopolymers containing triethanolamine are more responsive to moisture than photopolymers containing *N*-phenylglycine and display significant sensitivity to relative humidity above 40%. Full reversibility of humidity induced changes in gratings recorded in diacetone acrylamide-based photopolymer is confirmed at  $RH \le 60\%$ . Exposure to  $RH \ge 70\%$  leads to irreversible changes in these gratings.

The temperature response of slanted transmission gratings was investigated in the temperature range of 20-60 °C. Exposure of the photopolymer layers containing triethanolamine to elevated temperature was found to cause layer shrinkage due to desorption of absorbed water. Sealed layers containing triethanolamine, however, demonstrated swelling due to the effect of thermal expansion. The photopolymer layers containing *N*-phenylglycine were found to be unresponsive to temperature changes below  $30 \degree$ C and have sensitivity to temperature above  $30\degree$ C.

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#### 1. Introduction

Holographic gratings capable of responding to an analyte with an easily identifiable change in their optical properties have generated wide interest due to their possible application as holographic sensors. It has been demonstrated that holographic sensors can detect analytes in gas and in liquid phase as well as different physical stimuli such as pressure and magnetic fields [1–12]. Holographic sensors are considered a low-cost, lightweight, and disposable technology and have potential for application in different areas ranging from medical diagnostics to environmental sensing including the monitoring of environmental temperature and relative humidity [10,12,13]. In spite of the existing wide range of tem-

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http://dx.doi.org/10.1016/j.snb.2016.08.052 0925-4005/© 2016 Elsevier B.V. All rights reserved. perature and humidity sensors [14,15], holographic sensors are of special interest as they can provide fast, real-time, reversible or irreversible, visual colorimetric or optical readouts.

The intrinsic properties of some photopolymers, such as their hygroscopic nature and thermal expansion, make them good candidates for the development of humidity and temperature holographic sensors. Recently, the humidity sensitivity of reflection gratings recorded in an acrylamide-based photopolymer has been demonstrated and characterized in the range of 5–80% RH [10,12,13]. Reversible changes in fringe spacing due to water vapour absorption have been found to cause fully reversible variations in the diffracted light wavelength. The natural ability of the acrylamide-based photopolymer layer to swell or shrink at different levels of relative humidity was exploited for the design of a holographic humidity indicator [10].

The response to humidity of unslanted transmission gratings in similar material has been investigated in the range of 20–90%







RH with particular emphasis on irreversible changes caused by high humidity (RH  $\ge$  80%) [16]. Exposure to high humidity was found to cause changes in both the diffraction efficiency and the Bragg selectivity curve of these holographic transmission gratings. Reversibility of the observed changes at high humidity strongly depended on temperature and was confirmed for temperatures below 15 °C. The temperature limit of reversible/irreversible changes was established to correlate with the freezing/melting temperature range of one of the photopolymer components – triethanolamine. The ability of the transmission gratings to respond to high humidity is beneficial for the development of irreversible humidity holographic sensors, in particular for scenarios where increased temperature is also problematic e.g. food storage.

The humidity induced changes in the properties of both transmission and reflection slanted gratings recorded in an acrylamide based photopolymer has been quantitatively analysed [17] and the theoretical model of swelling dynamics has been proposed [18].

In this study the capability to tune the temperature and relative humidity response of a holographic grating by compositional changes in the photopolymer layer has been explored. Two main components have been varied - the type of monomer (acrylamide versus diacetone acrylamide) and the type of initiator (triethanolamine versus *N*-phenylglycine). The two monomers were chosen because it was previously observed that gratings recorded in photopolymer layers containing acrylamide and diacetone acrylamide have very different holographic and mechanical properties [19] which can influence on humidity and temperature sensitivity of the material. The two initiators were chosen because it was previously found that photopolymer layers containing triethanolamine are less dense and more permeable to water vapour and as a result the properties of photopolymer-based holographic gratings are humidity-dependant [16,17]. Whereas, photopolymer layers containing N-phenylglycine are robust and the photopolymer-based holographic gratings are irresponsive to humidity changes up to RH = 70% [20,21].

The first aim of this work was to study the humidity sensitivity of volume phase slanted gratings recorded in a non-toxic and environmentally compatible photopolymer containing diacetone acrylamide as the main monomer and triethanolamine as a photoinitiator. Comparative analysis of the humidity response of diacetone acrylamide-based gratings and acrylamide-based gratings has been carried out. Application of the diacetone acrylamide-based photopolymer as a humidity-responsive material for the development of holographic humidity sensors is also discussed.

As known, photopolymers in general have a low glass transition temperature and a relatively large coefficient of thermal expansion. Temperature variation induces grating detuning effects via changes in refractive index and the physical dimensions of the photopolymer layers [22]. Photopolymers that exhibit a mechanical response to temperature change are required for holographic temperature sensor development. Volume changes with temperature may be implemented as a sensing mechanism in holographic temperature response of acrylamide-based photopolymers are limited. The investigation of the reflection grating response to temperature in the range of 15–60 °C showed that the temperature response highly depends on the relative humidity level [13].

The second aim of this work was to study the temperature sensitivity of volume phase slanted gratings recorded in photopolymer containing either acrylamide or diacetone acrylamide as monomer and either triethanolamine or *N*-phenylglycine as an initiator. The potential application of photopolymers as a thermo-responsive material for the development of holographic temperature sensors is discussed.

#### 2. Background

There are two types of holographic sensors depending on the recording geometry of the holographic grating utilized. The first type is a sensor based on a transmission holographic grating. This sensor can change its transmittance when interaction with a target analyte occurs. The sensor requires both a light source for its illumination and a photodetector to monitor a change in a signal level, such as a diffraction efficiency alteration or a variation of the diffracted light direction.

A volume phase transmission grating can be recorded using two laser beams when the photosensitive medium is placed in the region of overlap of two interfering wavefronts which are incident on the photosensitive medium from the same side. The interference pattern created by two light waves is recorded as a spatial modulation of the refractive index of the photosensitive medium. The volume phase grating regime corresponds to Q » 1, which is defined by the relation [23].

$$Q = \frac{2\pi\lambda d}{n\Lambda^2},\tag{1}$$

where  $\lambda$  is the wavelength of the recoding light, *d* is the thickness of the grating, *n* is the average refractive index of the medium and  $\Lambda$  is the fringe spacing. The diffraction efficiency ( $\eta$ ) of a volume phase transmission grating at Bragg incidence is determined by the coupled wave theory [24].

$$\eta = \sin^2 \left( \frac{\pi n_1 d}{\lambda \cos \theta} \right),\tag{2}$$

where  $n_1$  is the refractive index modulation,  $\lambda$  is the wavelength of the reconstructing beam,  $\theta$  is the Bragg angle inside the photopolymer layer.

Exposure to an analyte leads to diffraction efficiency alterations  $(\Delta \eta)$  caused by the effect of different parameters which can be determined by the differentiation of Eq. (2) [3].

$$\frac{\Delta\eta}{\eta} = \left| \frac{2}{\tan\left(\frac{\pi n_1 d}{\lambda \cos\theta}\right)} \frac{\pi n_1 d}{\lambda \cos\theta} \right| \left( \frac{\Delta n_1}{n_1} + \frac{\Delta d}{d} - \frac{\Delta\lambda}{\lambda} + \tan\theta\Delta\theta \right), \quad (3)$$

where  $\Delta n_1$  is the refractive index modulation change,  $\Delta d$  is the thickness change,  $\Delta \lambda$  is the variation of probe wavelength and  $\Delta \theta$  is the Bragg angle shift.

The second type of holographic sensor is a sensor based on a reflection grating. The sensor operates via changes in the wavelength (colour) of the diffracted light under exposure to an analyte. These sensors are a focus of research as they can be used as visual indicators which can be easily interpreted by non-specialist. The reflection grating is recorded when two wavefronts reach the photosensitive medium from opposite sides. The direction of the maximum intensity of the light diffracted from the periodic photonic structure is determined by Bragg's law

$$2n\Lambda\sin\theta' = \lambda,\tag{4}$$

where  $\theta'$  is the Bragg angle which is defined by the angle between the incident beam and the planes of varying refractive index recorded in the material. Differentiation of Eq. (4) allows evaluating the contribution of different parameters on the spectral peak position of the diffracted light [3].

$$\frac{\Delta\lambda}{\lambda} = \frac{\Delta n}{n} + \frac{\Delta\Lambda}{\Lambda} + \cot\theta' \Delta\theta'.$$
(5)

Bragg angles in reflection geometry of recording are typically close to  $90^0$ . Thus, the contribution of the last term in Eq. (5) is usually negligible.

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