



Effect of zeolite nanoparticles on the optical properties of diacetone acrylamide-based photopolymer



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ABSTRACT

The effect of zeolite nanoparticles with BEA type framework structure on the optical properties of a diacetone acrylamide (DA)-based holographic photopolymer has been investigated. Both types of zeolite nanocrystals, as synthesized (BEA) and silver containing (BEA-Ag), are used for doping the DA photopolymer. Doping of the DA photopolymer with BEA zeolites results in an 11% decrease of the average refractive index of the nanocomposite layer, while the inclusion of BEA-Ag nanoparticles decreases the average refractive index by 3% only. This observation is in good agreement with the fact that the contrast in refractive index between the host photopolymer and nanoparticles is expected to be much lower for the BEA nanoparticles containing Ag in their pores. Doping of the DA photopolymer with pure BEA zeolite nanoparticles results in a significant increase in refractive index modulation due to holographic recording, Δn , of up to 91% at a concentration of 2 wt.% compared to un-doped layers. For the BEA-Ag zeolites, a maximum increase in Δn of up to 17% at a concentration of 0.5 wt.% is observed. The increase in Δn is attributed to the difference in n between the host photopolymer and nanoparticles and the redistribution of the nanoparticles during holographic recording. It is demonstrated that the DA photopolymer is compatible as a host material for the porous BEA nanoparticles. This is due to the large size of the DA monomer molecules, which restricts monomer migration into the zeolite pores and thus the pores of the Ag-free BEA nanoparticles remain empty, making them suitable for application in holographic sensors.

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1. Introduction

Photopolymers are fast becoming one of the most popular recording media for different holographic applications. Their high sensitivity, wide dynamic range and self processing nature, together with their low cost of production, make them an excellent candidate for applications such as holographic sensors, diffractive optics and data storage [1–9]. Large refractive index modulation (Δn) of the photopolymer material is favourable for holographic applications, in order to obtain high diffraction efficiency in thinner layers, and increased data storage density.

The idea of incorporating inorganic nanoparticles into holographic photopolymers was first introduced in 1996 by Oliveira et al. [10]. By selecting nanoparticles with a refractive index significantly different to that of the host photopolymer material, large Δn values can be achieved due to spatial redistribution of the nanoparticles during holographic recording (Fig. 1). Aside from large Δn , other positive effects due to their addition include reduced photopolymerisation-induced shrinkage, increased sensitivity, and improved grating stability. Problems with these nanocomposites can arise due to incompatibility of the nanoparticles with the photopolymer material, as well as increased optical losses due to scattering, if the difference between the refractive index of the nanoparticles and host material is too great [11,12].

Here, the effect of zeolite nanoparticles with BEA type structure on the optical properties, namely average refractive index (n) and refractive index modulation during holographic recording (Δn),

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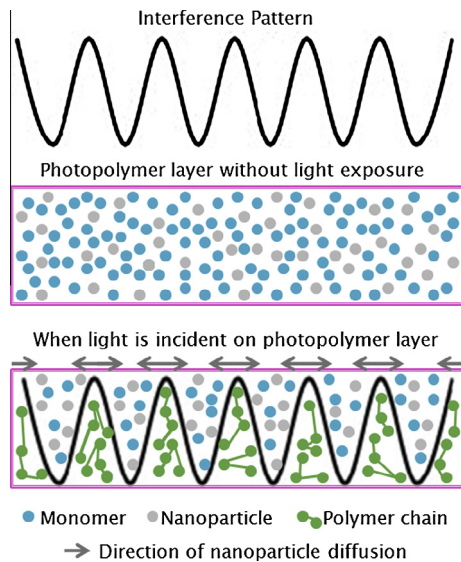


Fig. 1. Schematic diagram of grating formation in nanoparticle-doped photopolymer layer on exposure to light.

of a low-toxicity Diacetone Acrylamide (DA)-based photopolymer formulation is reported. The holographic characterisation and cytotoxicity evaluation of the DA photopolymer are reported elsewhere [13–15].

The incorporation of nanoparticles into holographic photopolymers has been extensively researched by many groups for a wide range of inorganic nanoparticles. Oliveira et al. observed that the diffusion of ZrO_2 ($n = 2.1$) nanoparticles within a methacrylate photopolymer resulted in Δn values of up to 1.5×10^{-2} [10]. A description of the generic technique of holographic patterning using nano and mesoparticles was published in 2001 by Vaia et al. [16]. The incorporation of TiO_2 nanoparticles (15 nm diameter, $n = 2.55$) in an acrylate photopolymer formulation by Suzuki et al. led to Δn values of 5.1×10^{-3} [17]. Reduced shrinkage was also observed. Sanchez et al. improved on this result by using 4 nm diameter TiO_2 nanoparticles, reaching Δn values of 15.5×10^{-3} [18]. In 2005, Tomita et al. verified that the significant improvements observed in Δn was due to the mass transport of the nanoparticles during holographic recording, with spatial redistribution of the nanoparticles occurring from bright to dark fringe regions [19]. Other inorganic nanoparticle types with different n values such as SiO_2 ($n = 1.46$) [20,21] have been investigated also, and similar improvements in Δn are reported. In 2008 Tomita et al. demonstrated that the inclusion of ZrO_2 and SiO_2 nanoparticles in (meth)acrylate photopolymers prevents thermal distortion effects, thus making them suitable for holographic applications over a wider range of environmental conditions [22].

Other metal nanoparticle types such as Au and Ag have been reported to cause an increase in Δn due to redistribution. Goldenberg et al. reported Δn values of 7.3×10^{-3} for a 1.5 wt.% Au-doped acrylate photopolymer [23]. Xue et al. state that the dispersibility and solubility of Au nanoparticles in acrylate formulations is greatly improved by coating the nanoparticles with citrate ions, due to their electrostatic repulsion action [24]. Research has been carried out into the in situ fabrication and redistribution of Ag nanoparticles by Balan et al. in methacrylate-based photopolymer formulations [25–27]. The 5 nm diameter Ag nanoparticles are observed to form due to the reduction of silver cations in the photopolymer solution on exposure to light. Pramitha et al. observe Δn values of 7.5×10^{-3} for their Ag-doped, water-soluble, acrylate photopolymer using this method [28].

Holographic polymer-dispersed liquid crystal devices (H-PDLCs) combine the advantages of photopolymer media with that of photonic crystals. Holographic patterning of these materials produces switchable devices, an effect which has interesting application for holographic optical elements [29]. H-PDLC materials are reported to achieve diffraction efficiencies greater than 90% and Δn values of up to 1.8×10^{-2} in 23 μm layers [30].

Different quantum dot nanoparticles such as CdSe [31] and ZnO [32] have been incorporated into photopolymer formulations due to their high fluorescence at certain wavelengths. This property is particularly interesting for holographic applications such as sensing and product authentication.

Acrylamide (AA)-based photopolymer nanocomposites containing three different zeolite nanoparticles with varying microporosity, namely Silicalite-1 (MFI-structure, diameter of 30 nm), AlPO-18 (AEI-structure, diameter of 180 nm) and Beta (BEA-structure, diameter of 40 nm) have been investigated by Naydenova et al. [33–36]. It was determined that the hydrophobic nature of the MFI zeolites ensures its pores remain empty when incorporated in the host photopolymer, causing an increase in Δn when redistributed. The inclusion of MFI nanoparticles has also been reported to cause a decrease in shrinkage from 1.32% to 0.57% [37,38], which is just above the commercial limit of shrinkage for data storage applications [39,40]. No such improvement in Δn was observed for the AEI and BEA type nanoparticles. The AEI type zeolites are hydrophilic in nature, and thus contain water molecules in their pores. In the case of the large-pore BEA zeolites (approximate pore size of $7.6 \times 6.4 \text{ \AA}$) which are of interest here, it was discovered that the AA monomer molecules (approximate size of $5 \times 4 \text{ \AA}$) were migrating into the zeolite pores. In both cases the refractive index difference between the nanoparticles and the host polymer matrix is not large enough to cause significant increase of the refractive index modulation as a result of their redistribution. However Raman spectroscopy studies by Leite et al. reported that up to 40% of the BEA zeolites in the layer are redistributed during holographic recording, indicating that BEA nanoparticles in acrylate photopolymer formulations are highly suitable for holographic applications. It has already been demonstrated that the BEA-doped AA photopolymer can be used as a holographic sensor for toluene, due to changes in the grating optical properties on exposure to the gas [41].

As discussed above, the addition of BEA zeolite nanoparticles to the AA photopolymer material did not result in a large net change in n upon redistribution due to the migration of the AA monomer molecules into the zeolite pores. Therefore it is expected that by increasing the size of the monomer molecules in the host photopolymer material, migration of the monomer into the pores of the BEA zeolite will be prevented, thus achieving the maximum Δn possible. The DA monomer molecule is nearly twice the size of the AA monomer (approximate size of $10 \times 4 \text{ \AA}$). Therefore it was predicted that the DA-based photopolymer will show significantly larger improvement in Δn with the incorporation of the BEA zeolite nanoparticles than the AA-based photopolymer, as migration of the DA monomer molecules into the zeolite pores is restricted by the monomer size.

The effect of two different zeolite nanoparticles i.e. pure BEA and silver containing (BEA-Ag), on the optical properties of the DA photopolymer has been investigated: standard 'empty pore' BEA, and BEA whose pores have been loaded with silver nanoparticles of 1 nm diameter (BEA-Ag). By loading the zeolites with Ag in this way, the n of the final particle is changed, allowing for comparison of the effect of nanoparticles with different n values on the DA photopolymer without altering the structure of the nanoparticle itself.

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