

Article

A Facile Fabrication Route of Poly(Ethylene Glycol Phenyl Ether Acrylate) Photopolymers with Efficient Optical Response for Holographic Storage

Fang-Yong Lee , Xin-Jin Wang, Wei-Hung Su and Tzu-Chien Hsu *

Department of Materials and Optoelectronic Science, National Sun Yat-sen University, Kaohsiung 804, Taiwan; gn0137exe@gmail.com (F.-Y.L.); jim40929@gmail.com (X.-J.W.); wxs156@mail.nsysu.edu.tw (W.-H.S.)

* Correspondence: tjhsu@mail.nsysu.edu.tw

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Abstract: A series of photopolymers based on ethylene glycol phenyl ethyl acrylate (EGPEA) monomers and poly(methyl methacrylate) (PMMA) matrix with varying initiator concentrations and sample thicknesses have been synthesized and their optical performance characterized in this study. The advantages of lowering the initiator concentration, including a rather short initiation time within a few seconds; a sharp rising optical response; and a stable saturated diffraction efficiency are demonstrated. The variation in the diffraction efficiency and response time with the exposure energy under various sample thickness and initiator concentrations is examined; a diffraction efficiency as high as 80% and a relatively short response time of 12–39 s are attainable. The dependence of the normalized optical parameter “sensitivity” on the exposure time is depicted, and the peak value of S ranges vastly from about 0.2 to 1.2×10^4 cm/J within a period of 15 s or so, with a maximum value of nearly 1.2×10^4 . Favorable evidence of low initiator concentration can further be found when the dependence of the saturated diffraction efficiency with the exposure energy is examined. The data from this study using a low initiator concentration cover a range of exposure energy from 100 to 800 mJ/cm² and a saturated diffraction efficiency from about 15% to 80%. The successful image reconstruction of 6-membered-ring metal nuts on a hologram based on this EGPEA/PMMA photopolymer system using a reflective holographic recording setup is demonstrated to verify its applicability to holographic storage.

Keywords: holographic storage; photo-initiator; diffraction efficiency; optical sensitivity

1. Introduction

Photopolymers have long been considered as crucial materials in developing technology related to photonic applications such as holographic data storage, holographic filters, holographic interferometry, and holographic optical elements [1–4]. Most holographic photopolymer systems are prepared by free radical polymerization, due to its relatively versatile fabrication process and highly efficient photo-initiators. Thick-film recording material (fulfilling Bragg’s condition) with a high optical quality is a key requirement among various applications. This condition cannot be easily achieved by traditional means of fabrication, because more optical scattering would happen whenever the thickness of the recording material is raised, diminishing the reliability of the holographic device [5,6].

In addition, volume shrinkage is regarded as another inevitable defect in the free radical-initiated photopolymers, caused by the rather rapid and uncontrollable radical reaction from the viewpoint of the polymerization mechanism. The shrinkage problem may be solved by adopting a pre-polymerized matrix binder with a matching refractive index to the polymerizing monomer used [7,8]. A 9,10-phenanthrenequinone (PQ)-doped methyl methacrylate (MMA) photopolymer with a matrix

binder PMMA was proved to be effective [2,5,9–11]. The refractive index uniformity throughout the whole holographic film was claimed to be primarily due to the minimized quantity of the residual monomers and photo-initiators involved in the fabrication of the photopolymer; also, the matrix structure was maintained during holographic recording [2,5,9–11]. Although the PQ/PMMA photopolymer system showed a great optical response and quality with the pre-polymerization method, its holographic recording at a widely used wavelength 532 nm was reported with a low sensitivity, which could be attributed to the fact that the resonance frequency of the PQ molecule is far away from 532 nm, thus reducing the hologram recording capability [12]. It is thus advised that the consistency of the optical performance between the absorption behavior of the absorption spectrometer and the holographic device should be carefully checked.

Other photo-initiators, such as bis-(2,6-difluoro-3-(1-hydropyrrol-1-yl)phenyl) titanocene 784 (Irgacure 784, or I-784), have been frequently used in holographic applications. The optical performance at 532 nm green light for I-784 is better and more efficient than PQ, as far as the optical response, the absorption coefficient, the resonance frequency, and the optical density are concerned [12,13]. The formation of holographic recording in the photopolymer system is generally attributed to the photo-initiator-induced free radical polymerization of monomers, and they form a phase grating. Major polymerization occurs in the constructive interference area (bright region). The photo-initiator and monomer molecules diffuse from the region of destructive interference area (dark region) into the bright region; consequently, a difference between the refractive indices in the dark and the bright regions is created, and thereby a phase grating is formed [6,14,15].

The initiator concentration has played an instrumental role in deciding the optical performance of the related photopolymers for holographic storage. This can be verified by the two well-known and well-developed systems—i.e., PQ/PMMA and I-784/PMMA. The concentration of PQ and I-784 has been limited by solubility, with the PQ being only 0.7 wt % and I-784 up to 7 wt % [13]. The latter is 10 times greater than the former; however, the optical performance of I-784 has been not as good as it should be as far as solubility is concerned. In practice, the widely used photo-initiator concentration in related photopolymer systems has been about 1–10 wt %, which gives a reasonable optical sensitivity and response [12]. From the viewpoint of better phase grating formation in the holographic film and the better crack-resistance of the photopolymer itself, a commercially available and widely used monomer ethylene glycol phenyl ethyl arylate (EGPEA) was chosen due to its high refractive index and more rigid poly(ethylene glycol phenyl ethyl acrylate) (PEGPEA) that was thus formed.

Sabol et al. studied the parameters influencing the optical characteristics of photopolymers and established a model of photo-initiation kinetics with a varying exposure power density (mW/cm^2) and I-784 concentration at 532 nm [16]. Based on Sabol's results, an attempt to lower the photo-initiator concentration was made in this study in order to maximize the efficiency of the optical response of PEGPEA. Therefore, a concentration of I-784 added into the system was controlled from 0.37 to 0.41 wt % (relative to EGPEA), while the solvent and PMMA matrix remained unchanged. Thus, the I-784 concentrations were at roughly one-twentieth of the reported maximum solubility, and were far less than the widely used concentrations in many photopolymer studies [1,2,5,9–13].

2. Materials and Optical Measurement

All the materials in this work were used as received. PMMA (molecular weight 300,000 g/mole, refractive index 1.490) and EGPEA (molecular weight 100.12 g/mole, refractive index 1.518) were purchased from Sigma-Aldrich (St. Louis, MI, United States); solvent acetone (A.C.S. grade, purity >99.5%) was purchased from Baker. PMMA as the matrix for EGPEA was dissolved in excess acetone with mechanical stirring at room temperature for 24 h. Part of the acetone would evaporate during this stage, yet the solution remained viscous. It was then mixed with EGPEA monomers and the yellowish photo-initiator I-784 (Ciba) by vigorous mechanical stirring for 1/2 h. The weight ratios of EGPEA to PMMA were kept at 0.59, 0.65, and 0.69 for the P-1, P-2, and P-3 series, respectively. This yellowish viscous solution was then cast into a glass substrate to form a holographic thin film, which was circled

by an O-ring rubber. The thickness of the thin film, ranging from ca. 100 to 500 μm could be controlled by adjusting the amount of acetone. This thin-film holographic material was then placed in dark at room temperature for an additional 24 h to ensure that all the acetone was completely removed. A series of experiments was performed to evaluate the optical sensitivity and diffraction efficiency of the holographic film with various film thicknesses and I-784 concentrations.

Figure 1 shows the holographic setup for the holographic recording and reading. In the recording process, a DPSS 532 nm laser was used as the light source. A beam splitter (BS) divided the laser beam into two parts with the same power and polarization mode. These two beams were interfered in the holographic medium with an incident angle of 3° (θ_1) and a total power density of 12.55 mW/cm^2 . In the reading process, a He-Ne laser with a wavelength of 633 nm was used to measure the diffraction efficiency η . Based on Bragg's condition, the incident angle of the reading beam was approximately 3.6° (θ_2). In this study, η is calculated by the ratio of I_d/I_t , where I_d is the 1st order intensity and I_t is the intensity of the incident light before interference at 633 nm. A power meter was used to measure the laser intensity in a continuous recording mode with about a 3–4 Hz sampling rate.

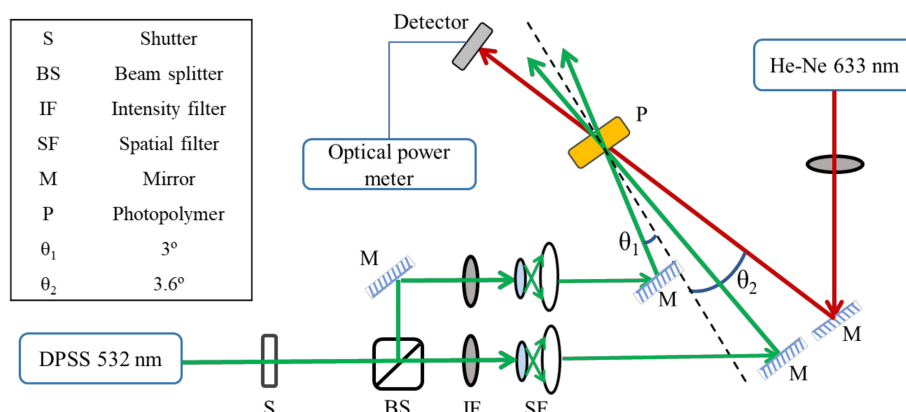


Figure 1. Holographic inscription and reading setup. Beams are colored by green and red for inscription and reading, respectively.

3. Results and Discussion

3.1. Optical Efficiency of EGPEA/PMMA Photopolymers

A typical diffraction efficiency behavior from one of the P-2 series in this study is shown in Figure 2. A relatively short initiation time is generally required, during which free radicals are generated from the cleavage of I-784 (Period I). This is followed by a steep rise in the diffraction efficiency, due entirely to the very fast photopolymerization caused by the sequential addition of EGPEA monomers to the I-784 free radicals (Period II). In Period III after the rising, a very smooth and saturated plateau is finally reached, defined hereafter as η_{sat} . The optical behavior shown in Figure 2 resembles pretty much the behavior of the typical free radical polymerization. This, in turn, provides a strong dependence of the optical performance on the polymerization efficiency. On the other hand, the shrinkage problem generally encountered in the free radical polymerization should be addressed here. Since the samples prepared in this study are relatively thin and the incident green laser light impinges on the sample only within a tiny spot 6 mm in diameter and with an estimated total input energy into the exposure area of 56 mJ, the thermal shrinkage during the irradiation should be insignificant. The beneficial effect of lowering the initiator concentration on the optical performance include a rather short initiation time (in a few seconds), a sharp rise in diffraction efficiency (with a few seconds too), and a stable and saturated plateau. A detailed comparison to the literature data and discussion can be found later in this section.

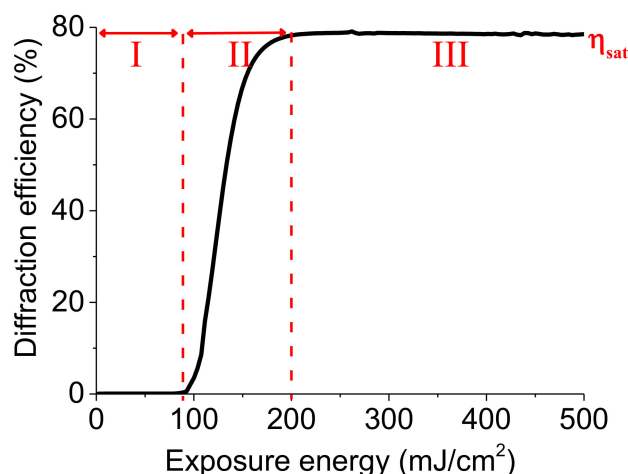


Figure 2. Typical diffraction efficiency behavior (sample P-2, with thickness 220 μm).

The variation in the diffraction efficiency with the exposure energy for the P-2 series under various sample thicknesses is shown in Figure 3a. Two parameters are adopted to evaluate the optical performance: (1) the saturated diffraction efficiency η_{sat} and (2) the response time t_{sat} to achieve the saturated diffraction efficiency. In this series, the diffraction efficiency tends to increase with increasing sample thickness, even though it fluctuates within those thinner samples, but still within a reasonable range. A diffraction efficiency as high as 80% is attainable. Additionally, the response time to reach the equilibrium diffraction efficiency is within a relatively short time, ranging from 12 to 39 s. The response time also tends to decrease with a decreasing sample thickness, but again it fluctuates within those thinner samples. The detailed photo efficiency data for the P-2 series, along with the other P-1 and P-3 series, are summarized in Table 1. The same effect of sample thickness on the diffraction efficiency for the P-3 series can be found therein.

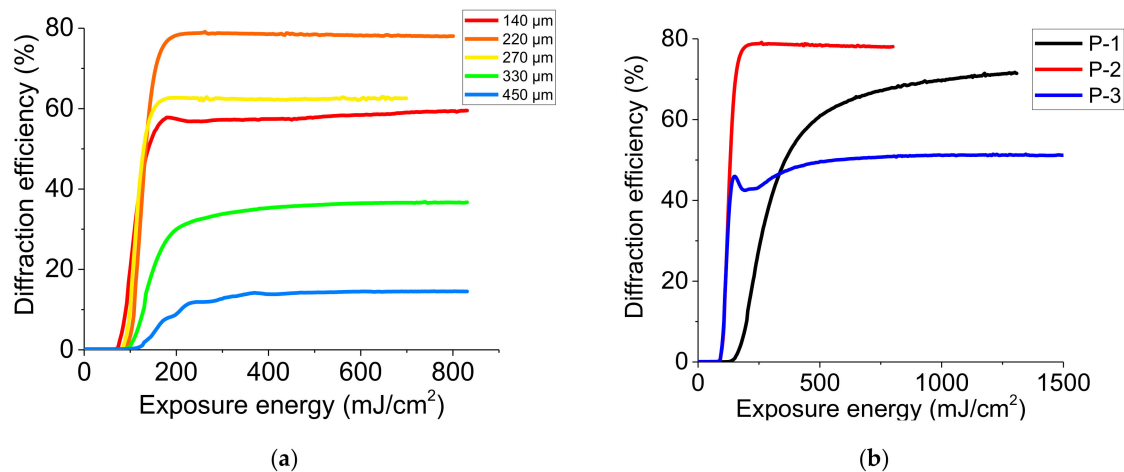


Figure 3. (a) Variation in the diffraction efficiency with exposure energy for the P-2 series with various sample thicknesses; (b) representative diffraction efficiency vs. exposure energy at three initiator concentrations. Sample thicknesses for P-1 to P-3 is 230, 220, and 230 μm , respectively.

Table 1. Diffraction efficiency and response time at saturation under varying photo-initiator concentrations.

Sample	I-784 (wt %)	Thickness (μm)	η_{sat} (%)	t_{sat} (s)
P-1	0.37	110	24	46
		140	27	61
		230	68	70
		460	74	24
		520	56	20
P-2	0.39	140	57	14
		220	80	16
		270	63	12
		330	34	39
		450	14	28
P-3	0.41	100	58	15
		170	42	54
		230	48	30
		380	61	21
		520	33	13

The effect of the sample thickness on the optical performance could be reasoned from the photopolymerization mechanism. Excess monomers would cross the boundary from destructive into the constructive interference area and minimize the refractive index difference. The migration of the monomers induces a mass transport between the constructive and destructive interference areas, thus resulting in a surface relief grating (SRG). Yet, the SRG of the free radical polymerization-based holographic medium is generally not as significant as those azobenzene-based ones (few nm vs. hundred nm). Therefore, it is normally ignored in most cases. For a thick sample, it results in a reduced diffraction efficiency and a prolonged response time, since it is more difficult to transmit the incident green light at 532 nm. The low transmission of the thick sample could be the main reason for the poor optical performance and thus the low diffraction efficiency. As for the absorption at 532 nm, it is the initiator I-784 which controls the absorbance throughout the whole media, and this is nothing to do with the polymerizing monomers or the matrix. In the beginning, the initiators absorb the green light to generate free radicals; these free radicals then “ignite” the instantaneous polymerization of EGPEA monomers. On the other hand, there should not be any absorption at 632 nm during the reading stage. The thickness effect can also be argued from the viewpoint of Rayleigh scattering. When a light beam propagates through the EGPEA/PMMA medium in which there are small growing polymeric particles, it becomes scattered with a decayed intensity and attenuated in the direction of propagation. A thicker sample with more growing particles thus scatters more. Furthermore, the scattered light inside the medium would interfere with each other, because each individual particle acts as a source of spherical wave. Therefore, the reading beam spends part of energy to reconstruct such a spherical wave and the energy of the 1st order diffraction is weakened for a thicker sample, resulting in a reduced diffraction efficiency.

The interference period is reduced as the incident angle θ_1 is increased. The reduced grating period further causes more monomers to overflow from the constructive to the destructive interference area. Hence, the difference in refractive index is reduced, leading to a weak diffraction. The diffraction efficiency could be close to zero, as the incident angle θ_1 is increased to 50° . Based on Bragg’s law, the equivalent holographic resolution could be computed. With an incident green light 532 nm at incident angle 3° and a refractive index 1.5 of the PMMA matrix in this study, a holographic resolution of 296 lines/mm can be obtained. The resolution increases to 4323 lines/mm under an incident angle of 50° .

For all three initiator concentration series, the representative results of diffraction efficiency are shown in Figure 3b, where the diffraction efficiency falls between 48% and 80%, and the response

time is from 16 to 70 s. With almost nearly equal sample thicknesses, the results suggest a better optical performance at a higher initiator concentration. For the highest P-3 series in particular, the low diffraction efficiency and prolonged response time may be attributed to relatively low light transmittance, since the yellowish I-784 made the sample darker, allowing the sample to severely scatter light. Thus, the efficiency of the optical response is significantly reduced.

The above results indicate that the optical performance of a holographic recording is profoundly affected by the sample thickness, along with the intensity of the incident light. In order to avoid the confusion, a “normalized” parameter entitled “sensitivity (S)” as Equation (1), where the time derivative of the square root of the diffraction efficiency, was taken and further divided by the inscription beam intensity (I_0 , in W/cm^2) and sample thickness (d , in cm), is shown in the following [6]:

$$S = \frac{1}{I_0 d} \left(\frac{\partial \sqrt{\eta}}{\partial t} \right). \quad (1)$$

The dependence of S on the exposure time for P-2 series is presented in Figure 4, in which a peak S is generally found at a relatively short time within 10 s or so. The peak value of S ranges vastly from about 0.2 – to 1.2×10^4 cm/J within a period of 15 s or so, with maximum value of nearly 1.2×10^4 for the thinnest $140 \mu\text{m}$ sample. In other words, the thinner the sample thickness is, the higher the sensitivity. This provides evidence of the highly efficient optical performance of the holographic recording materials thus prepared.

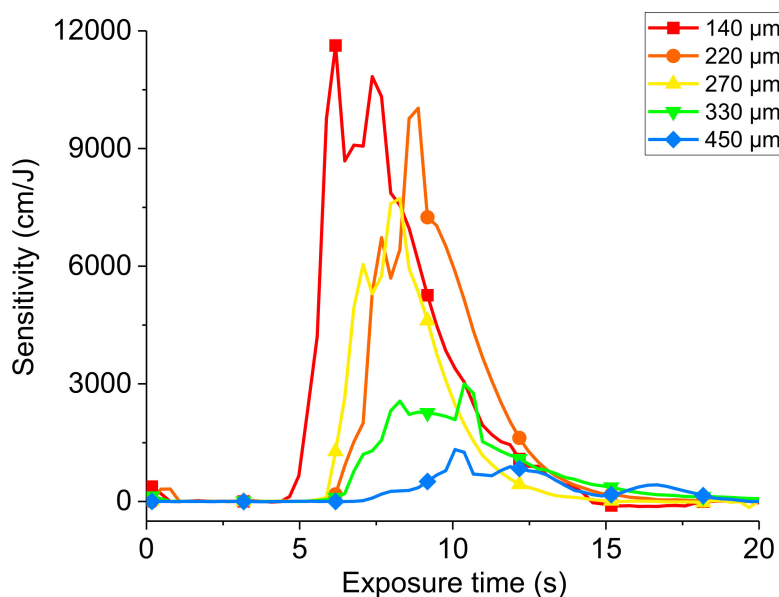


Figure 4. Dependence of S on the exposure time for the P-2 series.

To further illustrate the beneficial effect of the low initiator concentration, the optical performance of relevant literature is compared and summarized in Table 2. A comparison of data from this study to those made to data by Liu et al. indicates that a much higher exposure energy (200 vs. at least $32821 \text{ mJ}/\text{cm}^2$) is required, and also a much higher initiator concentration range (0.39 vs. 1–7%), yet with a smaller attainable saturated diffraction efficiency (80 vs. at most 52.2%) [13]. Although the diffraction efficiency from Oh et al. using a lower exposure energy is rather close to our findings, a shorter wavelength incident beam of 491 nm was adopted [17]. The comparison from Table 2 further provides evidence to support the beneficial effect of adopting a low initiator concentration for an efficient optical response of photo-initiated holographic storage material systems.

Table 2. Optical performance compared.

Monomer	Matrix	Wavelength (nm)	I-784 (%)	η (%)	Expo. Energy (mJ/cm ²)	Year/Ref
EGPEA + DT	PES	491	1.00	70.0	17.6	2008/[17]
MMA	PMMA	532	1.00	13.6	32821	2017/[13]
MMA	PMMA	532	3.00	32.9	54466	2017/[13]
MMA	PMMA	532	5.00	52.2	127323	2017/[13]
MMA	PMMA	532	7.00	52.2	127323	2017/[13]
EGPEA	PMMA	532	0.39	80.0	200	This study

Monomer DT = S-[4-(2-methylprop-2-enoylsulfanyl)-6-(N-phenylanilino)-1,3,5-triazin-2-yl] 2-methylprop-2-enethioate, MMA = methyl methacrylate; matrix PES = polysulfone, PMMA = poly(methyl methacrylate). I-784 concentration used in reference 13 was based on weight percent to monomer.

3.2. Holographic Image Recording on an EGPEA-Based Hologram

An optical setup based on reflection holography was built to evaluate the performance of 3D image reconstruction. Figure 5 depicts the configuration of the holographic recording. A specimen in P-2 series with thickness 220 nm and diffraction efficiency 80%, coated on a 2.5×2.5 cm glass substrate, was selected as the holographic material. The reference beam was a plane wave formed by launching the laser wave into a spatial filter and a collimated lens, while the object beam was produced by a diffusive reflection from two 6-membered-ring metal nuts (as shown in Figure 6a). The volume hologram was recorded with the reference and object beams arriving from opposite sides. Figure 6b shows the reconstructed 3D image by illuminating the reflection hologram with a white light source. It is evident that the profile of the original metal nuts is successfully recorded and holographically displayed on this EGPEA-based hologram. Considering that the whole holographic process was not digitally inscribed and without using a multiplexing technique, the quality of this reconstructed holographic image was not as clear as expected. This necessitates the further improvement of the optical setup in the inscription conditions.

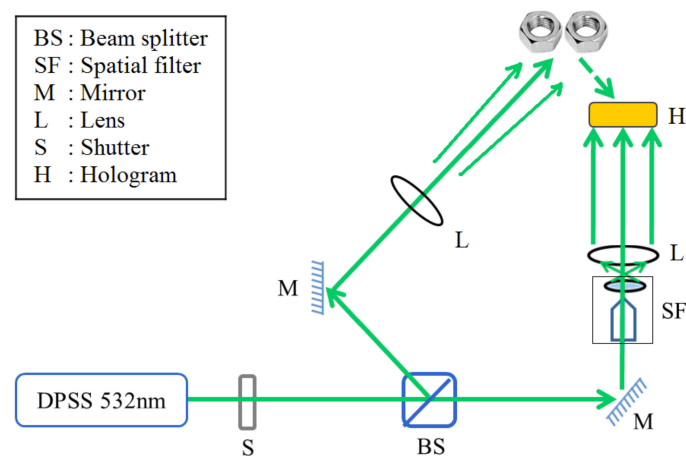


Figure 5. Holographic inscription setup for recording and reconstructing the image of two 6-membered-ring metal nuts on the EGPEA-based hologram.

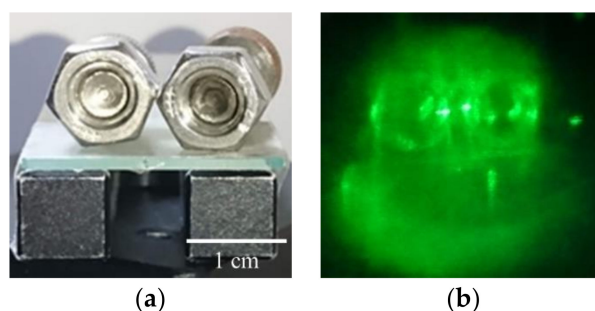


Figure 6. (a) The appearance of the metal nuts, and (b) the reconstructed 3D image from the EGPEA-based hologram.

4. Conclusions

The effect of a low initiator concentration on the optical performance of EGPEA/PMMA holographic storage materials has been demonstrated in this study. The advantages of lowering the initiator concentration include a rather short initiation time (in a few seconds), a sharp rise in the diffraction efficiency (with a few seconds too), and a stable and saturated plateau. The study of the variation in diffraction efficiency with the exposure energy for an EGPEA/PMMA under various sample thicknesses and initiator concentrations indicates that the diffraction efficiency increases with a decreasing sample thickness; the thinner the sample thickness, the higher the diffraction efficiency. A diffraction efficiency as high as 80% and a relative short response time of 12–39 s are attainable. The response time decreases with a decreasing sample thickness; the thinner the sample thickness, the faster the response time. A “normalized” parameter called “sensitivity (S)” was used to eliminate the influence of the sample thickness and the intensity of the incident light. The study of the dependence of S on the exposure time shows that the peak value of S ranges vastly from about 0.2 to 1.2×10^4 cm/J within a period of 15 s or so, with a maximum value of nearly 1.2×10^4 . The thinner the sample thickness is, the higher the sensitivity. Favorable evidence of a low initiator concentration can further be seen from the dependence of the saturated diffraction efficiency on the exposure energy. Our data cover a range of exposure energy from 100 to 800 mJ/cm² and saturated diffraction efficiencies from about 15% to 80%, as compared to the literature data, with an exposure energy 10 times higher and a lower diffraction efficiency when under a lower exposure energy. Subsequent image reconstruction on a hologram based on this EGPEA/PMMA photopolymer system using a reflective holographic recording setup was followed to verify its applicability to holographic storage. The holographic image of the original 6-membered-ring metal nuts was successfully reconstructed on this EGPEA-based hologram.

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