High-frequency relief holographic gratings created by exposing dichromated gelatin to short-wave UV radiation

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Abstract. The possibility of creating effective relief holographic gratings with a high spatial frequency of 1500 mm⁻¹ on the layers of dichromated gelatin (DCG) using the destructive effect of short-wave UV radiation on gelatin is shown for the first time.

1. Introduction

There is a method for converting amplitude holographic recording into relief-phase on silver halide photoemulsions based on photolytic effect of short-wave UV radiation with a wavelength of less than 250 – 270 nm on gelatin (SWUV method) [1, 2]. Subsequently, this method was applied to the DCG layers to create low-frequency diffraction gratings with a spatial frequency ν of the order of 100 – 200 mm⁻¹ [3]. The heights of the surface relief of more than 1 μm obtained by this method make it possible to create on the DCG layers a various random and regular structures, operating in a relatively small range of diffraction angles of the order of 10-15°.

It is possible to significantly expand the scope of the use of relief holographic structures on the layers of DCG with increasing spatial frequency, since spatially periodic structures with frequency ν ≥ 1000 mm⁻¹ are widely used as diffractive optical elements, antireflection surfaces, filters, modulators, etc. [4-8].

2. Features of the use of shortwave UV radiation when creating surface relief on the DCG layers

The steps for obtaining surface relief on the DCG layers in the case of using UV radiation are shown in figure 1.

The change in the physicochemical properties of gelatin is carried out directly during the holographic recording of the interference pattern by the radiation of a He-Cd laser due to selective light tanning of gelatin in the presence of dichromates (step 1, figure 1a). In this case, structuring occurs, that is, a large number of cross-links between gelatin molecules are established at the maxima of the interference pattern. Primary processing of samples after exposing them by He-Cd laser, consisting of water bathing and drying (step 2, figure 1a), leads to the appearance of a relief height h of the order of 0.1 μm, insufficient to obtain a holographic structure with high diffraction efficiency (DE).
Figure 1. Stages of obtaining high surface relief on the DCG layers for high spatial frequency. (1)–(5) treatment steps. (a) standard treatment procedure involving prolonged (several minutes) bathing in water after UV irradiation of the sample. In this case, there is gelatin swelling over the entire thickness and as a result, smoothing of the surface relief during final drying. (b) modified treatment procedure in which a short bath in water (10 seconds) is interrupted by an isopropanol bath. This allows one to avoid swelling of the DCG inner layers and obtain a significant height of the surface relief during the final drying.

Laser-hardened areas of DCG are less susceptible to the damaging effects of short-wave UV radiation (step 3, figure 1a) since a large number of cross-links prevent fragmentation of gelatin macromolecules and their dissolution in water. Thus, the crests of the surface relief on the DCG layer after exposure to UV light and washing in water at room temperature and drying (steps 4, 5; figure 1a) are formed in the most tanned places, i.e. in the maxima of the interference pattern.

3. Creating holographic gratings with high spatial frequency

Previously, it was found [9] that gelatin-containing recording media for holography, regardless of the methods for obtaining relief-phase structures, have restrictions on the transmission of the space-frequency spectrum in the high frequency range $\nu \geq 200 \text{ mm}^{-1}$. This is reflected in figure 2 for the SWUF method.

Such a decline in the relief height $h$ and, consequently, in DE can be theoretically explained by the smoothing effect of surface tension forces arising in the wet gelatin layer at high spatial frequencies [9, 10]. This fact is illustrated in figure 1(a). A relatively long bathing samples in the water within a few minutes after UV radiation exposure according to the standard treatment used by us [3] causes the DCG layer to swell over the entire thickness. Therefore, soft supple gelatin is easily deformed under the action of surface tension forces, resulting in surface relief smoothing at high frequencies [9, 10] during final drying in air.

To decrease the influence of surface tension forces, it was proposed to significantly reduce the time of bathing samples in the water after UV irradiation (step 4, figure 1b). The short time of water treatment leads to the fact that the photodegraded irradiated areas of gelatin have time to dissolve in the water faster than the inner gelatin layers swell. Further use of the isopropanol bath (step 5, figure 1b) also prevents the layer from swelling in thickness. Therefore, only the surface layer of
gelatin is involved in the formation of the holographic structure, and the action of surface tension forces is minimized. Thus, in comparison with the standard technique the proposed one should lead to a significant increase in the surface relief height at high spatial frequencies (compare figures 1a, b).

Figure 2. The average dependence of the surface relief height $h$ on the spatial frequency $v$ for a SWUV method.

4. Experiment
DCG layers were produced under laboratory conditions similar to the known method described in [11]. Holographic gratings with spatial frequency $v = 1500 \text{ mm}^{-1}$ were recorded using a symmetrical optical scheme by He-Cd laser radiation with a wavelength of 440 nm. After exposure of the dry DCG layer, the grating samples were processed by bathing in a 2% sodium sulfite solution for 6 min to complete the gelatin tanning processes and remove chromium compounds from a layer.

After washing and drying, grating samples were illuminated by UV radiation using a high-pressure mercury-quartz lamp DRT-220 for 20-24 min. Then samples were processed for 10 s in the water and immediately immersed in pure isopropanol successively in two baths (1 min each) for rapid dehydration of the layer. Measurement of gratings DE was carried out by light of He-Ne laser with a wavelength of 630 nm at an optimal angle of incidence on the sample from the side of the gelatin layer.
Figure 3 (curve 1) shows the dependence of DE in the first diffraction order on the exposure time of the samples with a He-Cd laser. The achieved maximum DE 35% is many times higher than this value (1.73%) for the grating samples treated after UV irradiation according to the previously used standard technique [3] for the same high spatial frequency. Figure 3 contains curve 2, reflecting DE of areas of the same sample not exposed to UV radiation. Comparison of curves 1 and 2 shows a significant role of UV irradiation in the formation of deep surface relief due to the removal of destroyed gelatin molecules by water.

The presence of an effective relief grating is confirmed by experiments using an immersion liquid. Figure 4 shows the dependence of DE on the exposure time by coherent radiation for holographic gratings with an open surface (curve 1) and with a surface coated with immersion liquid closed with a cover glass on top (curve 2). Dill oil was used as an immersion liquid, the refractive index of which was approximately equal to the refractive index of gelatin. Dependences presented in figure 4 indicate a predominantly relief-phase mechanism of diffraction.

5. Conclusion
The efficiency of the used method of processing holographic structures on the DCG layers, characterized by a significantly short time of bathing samples in the water and the inclusion of isopropanol baths in the treatment for rapid dehydration of the layer, was shown. The new method of holograms processing using UV irradiation allowed for the first time to obtain relief-phase gratings with a high spatial frequency $\nu = 1500$ mm$^{-1}$ and a maximum DE 35% exceeding the values of the diffraction efficiency for the previously obtained low-frequency holographic gratings on the DCG layers.

The results of the work will contribute to the expansion of the application scope of relief holographic gratings recorded on such a widespread medium with high optical parameters as DCG.

References
[1] Gulyaev S N and Ratushnyi V P 2003 J. Opt Technol. 70 105
[2] Ganzherli N M, Gulyaev S N, Maurer I A, Chernykh D F and Yalovik S A 2012 Tech. Phys. 57 1230
[3] Ganzherli N M, Gulyaev S N and Maurer I A 2016 Technical Physics Lett. 42 988
[4] Onishi M 2011 Rigorous coupled wave analysis for gyrotropic materials Ph.D. dissertation College of optical sciences The university of Arizona Tucson 15
[5] Popov E 2012 Introduction to diffraction gratings: summary of applications // Theory and numeric applications (Marseille: Presses Universitaires de Provence PUP) pp. 1.1-1.23
[6] Bondos N and Neauport J 2016 Adv. Opt. Photon. 2016. 8 1–44
[7] Kniazkov A V 2018 Optical Memory and Neural Networks (Information Optics) 27 191-195
[8] Kniazkov A V 2017 Journal of Applied Physics 122 125106
[9] Gulyaev S N 2008 Surface relief formation of holographic structures produced by exposure of photographic emulsion to short-wave UV radiation SPbPU Journ. of Engineering Science and Technology 3(59) 105-14
[10] Butusov M M and Ioffe A I 1976 J. Quantum Electron 6 519
[11] Shankoff T A 1968 Appl. Opt. 7 2101-05