04

Improvement of the technology for manufacturing relief holographic gratings on dichromated gelatin irradiated with short-wave UV radiation

© N.M. Ganzherli[1], S.N. Gulyaev[2], I.A. Maurer[1]

[1] Ioffe Institute, St. Petersburg, Russia
[2] Peter the Great St.Petersburg Polytechnic University, 195251 St.Petersburg, Russia

e-mail: nina.holo@mail.ioffe.ru; gulyaev@rphf.spbstu.ru

Received on May 12, 2021
Revised on May 12, 2021
Accepted on July 09, 2021

A new variant of manufacturing high-frequency holographic gratings with a spatial frequency up to $1500\,\mathrm{mm}^{-1}$ on layers of dichromated gelatin (DCG), in the processing technology of which water procedures are completely excluded, is proposed. The method is based on the use of glacial acetic acid after the photodestruction of layers by exposure to short-wave UV radiation instead of the standard method of processing holographic structures registered by coherent He−Cd laser radiation. In the course of the experiments, relief-phase holographic gratings with high diffraction efficiency up to 64% were obtained, regardless of the thickness of the DCG layers.

Keywords: holographic diffraction gratings, dichromated gelatin, short-wave UV radiation, surface relief, diffraction efficiency, glacial acetic acid, isopropanol.

DOI: 10.21883/EOS.2022.13.53982.2283-21

Introduction

To obtain highly efficient relief-phase holographic gratings, including high-frequency gratings, as light-sensitive recording medium we used the layers of bichromated gelatin [1–6] made in laboratory conditions using a method similar to that described in [7]. The interference pattern is initially recorded in the form of modulation of the degree of layer tanning over the entire thickness when using He−Cd-laser radiation. The fundamental feature of the treatment is the irradiation of the layers with short-wave UV radiation of a mercury-quartz lamp with a wavelength of $\lambda < 270\,\mathrm{nm}$, which leads to photodegradation of gelatin in the least tanned places and, as a result, to its transfer to a soluble in water state [8]. The surface relief is formed due to washing out (etching) of the gelatin areas which are mostly destroyed by ultraviolet radiation. However, the presence of water treatment is a factor preventing the obtaining of the required depth of the surface relief at spatial frequencies of the holographic structure exceeding $200-300\,\mathrm{mm}^{-1}$ [9]. In a number of publications, we explained the reason for this fact by the influence of surface tension forces, which smooth out the surface relief in a wet swollen gelatin layer during drying [4,5,9].

Previously, we have demonstrated the methods of DCG layers treatment after irradiation with short-wavelength UV radiation, excluding both long-term water procedures leading to swelling [5], and water treatment in general, replaced by heat treatment of samples [6]. This allowed to obtain relief-phase holographic gratings with a spatial frequency of $1500\,\mathrm{mm}^{-1}$ and high diffraction efficiency (DE) exceeding 50% [5].

The book [10] gives the examples of organic solvents in which gelatin dissolves. They include acetic acid, glycerin, trifluoroethanol, formamide, dimethyl sulfoxide, ethylene glycol.

Previously, it was shown, that gelatin destroyed by UV radiation can be removed from the layer of silver halide photographic emulsion not only by bathing in aqueous solutions, but also by the effect of other factors on the photographic emulsion, for example, by glacial acetic acid (GAA) [9]. Experiments with GAA for low-frequency gratings with a spatial frequency of $110\,\mathrm{mm}^{-1}$ gave the surface relief height values, comparable with the values obtained by treatment in aqueous solutions according to the standard method [8]. According to [11], GAA is an effective gelatin solvent and, according to our observations, causes much less swelling of gelatin, than water. Therefore, it can be assumed, that for high-frequency gratings recorded on BCG layers, GAA is also an effective etching agent.

Experimental procedure

In the present study we studied a variant of treatment of high-frequency holographic gratings on DCG layers, in which, at the stage of formation of the surface relief of the holographic structure, the water procedure is completely excluded and replaced by the action of GAA or its solutions in isopropanol (isopropyl alcohol, IPA). Samples of DCG layers of different thickness were studied, fabricated under laboratory conditions, the treatment of which prior the exposure to ultraviolet is described in detail in the studies [1,2]. At the first stage, the gratings were registered by radiation from a He−Cd- laser with a wavelength of $\lambda = 440\,\mathrm{nm}$ and a spatial frequency of $1500\,\mathrm{mm}^{-1}$ according to a symmetric

optical scheme. At the same time, selective light tanning took place in DCG layers, i.e., structuring, which resulted in the formation of a large number of cross-links between gelatin macromolecules at the maxima of the interference pattern. Further bathing in 2% sodium sulfite solution for 6 min, including the removal of chromium compounds from the layer, completed the process of selective tanning. At the second stage the dried grating samples were exposed to UV radiation using a mercury-quartz high-pressure lamp DRT-220. The photostimulated degradation of gelatin under the action of high-energy photons of short-wave UV radiation with a wavelength of less than 270 nm includes a break in the bonds of the main chains of gelatin molecules. More tanned areas of the gelatin layer, corresponding to the maxima of the originally recorded interference pattern, are more resistant to the damaging UV radiation effect. Thus, the UV radiation effect on gelatin is also selective.

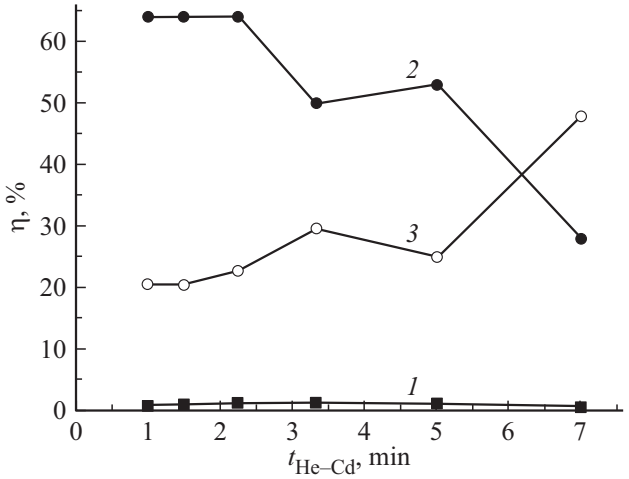

Figure 1. Dependence of DE gratings on the time of exposure of samples to radiation from a helium-cadmium laser. *1* — DE of the first diffraction order before treatment; *2* — DE of the first order after treatment; *3* — DE of zero order after treatment.

Results

In the course of experiments, the grating samples on DCG layers with a thickness of 1.1 to 13.5 μm, which was measured outside the region of recorded holographic gratings, were studied. The time of irradiation of the samples with short-wavelength UV radiation was 22−24 min. After the exposure to UV radiation, GAA was used to treat the layers in a different IPA percentage. The etching operation was carried out at room temperature. This was followed by washing the samples in two baths of 100% IPA for 1 min each and final drying in an air stream at room temperature.

In the course of the experiment carried out on a 13.5 μm sample using 100% GAA, masses of gelatin in the form of a whitish suspension were seen to be washed into the treatment bath. In this case, the DCG layer thickness decreased by almost a factor of 2. Therefore, to weaken the etching effect, it was decided to use further dilute GAA solutions in IPA.

Thus, a 5.65 μm sample was treated in a solution of 50% GAA+50% IPA for 13 s. Figure 1 shows the dependences of the DE η of the diffraction zero and first orders on the time of sample exposure to coherent radiation from a He−Cd-laser. The diffraction efficiency was determined as the ratio of the light intensity in the zero or first diffraction orders and the incident beam intensity. For reading, radiation from a He−Ne-laser with a wavelength $\lambda = 0.63\,\mu$m was used. The readout beam diameter was 2 mm. The samples were illuminated at an angle corresponding to the Bragg conditions. The maximum DE values (of the first order), equal to 64%, were observed for three gratings with different exposure times to coherent He−Cd- laser light.

The same GAA concentration in IPA and the same etching time were used to process a thinner sample — with a thickness of 1.65 μm. The values of DE gratings in this case were in the range of 12−18%, which is much lower, than the result obtained above. In this case, we associate

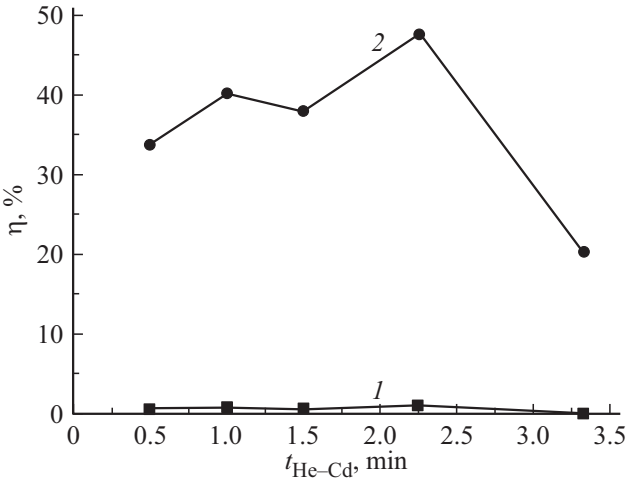

Figure 2. Dependence of DE gratings on the time of exposure of samples to radiation from a helium-cadmium laser for the sample with the thickness of 1.94 μm. *1* — DE of the first diffraction order before treatment; *2* — DE of the first order after treatment.

the decrease in the surface relief height, which determines the DE value, with excessive etching of a thin gelatin layer. Therefore, to treat thin samples, it was decided either to reduce GAA concentration in IPA, or to significantly reduce the etching time. The results of applying the proposed treatment options are shown in Fig. 2 and 3, which testify to the effectiveness of the use of the proposed modes of etching of thin DCG layers.

The parameters of the best samples of holographic gratings are summarized in the table. It shows, that high DE values can be obtained on the layers of even so small thickness, as 1−2 μm. A significant (up to 200 times) increase in DE after final treatment indicates the formation of a deep relief structure on the BCG layer surface.

Parameters of holographic gratings samples

Layer thickness DCG, μm	Concentration GAA in IPA, %	Time of etching, s	Initial DE of the 1-th order, %	DE of the 1-th order after treatment, %	Increase of DE, times
5.65	50	13	1.2	64	53
1.94	25	10	0.94	47.6	51
1.1	50	6	0.2	41.3	207

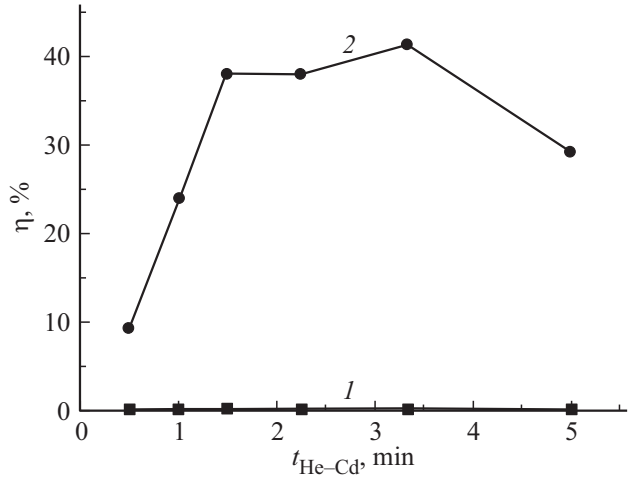

Figure 3. Dependence of DE gratings on the time of exposure of samples to radiation from a helium-cadmium laser for the sample with the thickness of 1.1 μm. *1* — DE of the first diffraction order before treatment; *2* — DE of the first order after treatment.

Conclusion

The effectiveness of the use of glacial acetic acid and its solutions in isopropanol in the technology of treatment of high-frequency holographic gratings with a high spatial frequency of 1500 mm^{-1}, recorded on BCG layers of different thicknesses, was demonstrated. The achieved values of the maximum diffraction efficiency (64%) exceed the best results obtained by us earlier in experiments with a short water treatment (55%).

Conflict of interest

The authors declare that they have no conflict of interest.

References

[1] *Ganzherli N.M., Gulyaev S.N., Maurer I.A.* // Techn. Phys. Lett. 2016. V. 42. N 10. P. 988. doi 10.1134/S1063785016100060
[2] *Ganzherli N.M., Gulyaev S.N., Maurer I.A.* // Opt. Spectrosc. 2018. V. 125. N 1. P. 65. doi 10.21883/OS.2018.07.46268.73-18
[3] *Ganzherli N.M., Gulyaev S.N., Maurer I.A., Khazvalieva D.R.* // Opt. Spectrosc. 2018. V. 124. N 3. P. 408. doi 10.1134/S0030400X18030116
[4] *Ganzherli N.M., Gulyaev S.N., Maurer I.A., Khazvalieva D.R.* // Techn. Phys. Lett. 2019. V. 45. N 12. P. 613. doi 10.1134/S1063785019060245
[5] *Ganzherli N.M., Gulyaev S.N., Maurer I.A., Arkhipov A.V.* // Optoelectronics, Instrumentation and Data Processing. 2020. V. 56. N 2. P. 77. doi 10.3103/S87566999020020065
[6] *Ganzherli N.M., Gulyaev S.N., Maurer I.A.* // Opt. Spectrosc. 2020. V. 128. N 10. P. 1618. doi 10.1134/S0030400X20100100
[7] *Shankoff T.A.* // Appl. Opt. 1968. V. 7. N 10. P. 2101. doi 10.1364/AO.7.002101
[8] *Gulyaev S.N., Ratushnyi V.P.* // J. Opt. Technol. 2003. V. 70. N 2. P. 105. doi 10.1364/JOT.70.000105
[9] *Gulyaev SN* Rel'efno-fazovye gologrammy na fotoemul'sionnyh sloyah, obluchennyh ul'trafioletovym izlucheniem (in Russian). Synopsis of PhD thesis. SPb, 2006. 196 p.
[10] *James T.H.* The Theory of the Photographic Process. NY.: Macmillan Publishing Co, 1977.
[11] *Umberger J.Q.* // Phot. Sci. Eng. 1967. V. 11. N 6. P. 385.