Technical Physics Letters, 2022, Vol. 48, No. 14

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Registration of high-frequency relief-phase holographic structures on the PFG-04 photographic material

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Received June 30, 2021
Revised July 14, 2021
Accepted July 15, 2021

The paper proposes a new variant of processing photographic plates for holography based on dichromed gelatin PFG-04 (produced by JSC „Slavich Company“, Pereslavl-Zalessky) for manufacturing high-frequency relief-phase holographic gratings with a spatial frequency of up to $1500\,\mathrm{mm}^{-1}$. The technology is based on the selective destructive effect of shortwave UV radiation on gelatin and subsequent etching of the layer with various reagents. The relief-phase high-frequency holographic gratings with the maximum diffraction efficiency of 67% were obtained on PFG-04 photographic plates for the first time.

Keywords: holographic relief-phase gratings, dichromed gelatin, photographic plates PFG-04, short-wave UV radiation, glacial acetic acid

DOI: 10.21883/TPL.2022.14.52103.18941

Photographic materials based on dichromed gelatin (DCG) along with haloid-silver photoemulsions are conventionally regarded as ones of leading holographic recording media because of their good optical parameters, high resolvability and diffraction efficiency of the recorded structures [1]. [1]. The procedure for processing the layers after hologram exposure, which is recommended by the plates manufacturer PFG-04, involves the layer dehydration successively in aqueous solutions of isopropyl alcohol 25, 50, 75 and 100% in concentration. This allowed obtaining relief-phase gratings with the diffraction efficiency (DE) of $\eta \approx 70\%$. In contrast to this conventional technique for fabricating relief holograms on DCG, in this study we have proposed and investigated a method for obtaining thin high-frequency relief-phase holographic structures based on the PFG-04 material. The method is based on the destructive action on gelatin by shortwave UV radiation with the wavelength of $< 270\,\mathrm{nm}$ [2] followed by etching the layer with different reagents. At the first stage, the gelatin was selectively tanned according to the interference pattern by recording holographic gratings $1500\,\mathrm{mm}^{-1}$ in spatial frequency with two flat He$-$Cd laser beams (with wavelength $\lambda = 0.44\,\mu\mathrm{m}$) using a symmetric optical scheme. Then photographic plates were „bathed“ in the 2% sodium sulfate solution, rinsed with water and dried, after which the samples were irradiated for 20$-$25 min with shortwave UV radiation of a mercury-quartz lamp DRT-220. The less hardened gelatin areas located in the interference pattern minima were stronger destructed by the UV radiation and, hence, were the first which were removed from the layer surface in subsequent etching. Thus, a deep surface relief was created for structures with the spatial frequency of up to $200-300\,\mathrm{mm}^{-1}$ [3].

For recording high-frequency diffraction relief gratings on DCG, an improved technique was proposed, which allows reduction of the effect of surface tension forces smoothing the wet layer surface relief in drying. The technique was characterized by a decrease in time of the layer staying in water to a few seconds [4], which led to reducing the gelatin layer swelling and to obtaining after etching and final drying a deep surface relief with high spatial frequencies. In this study, not only water was used for etching but also a 50% isopropanol solution of glacial acetic acid. Just as water, glacial acetic acid is an efficient solvent of gelatin [5,6], however, contrary to water, it does not cause considerable swelling of the DCG surface layers, which enables persistence of the deep relief at high spatial frequencies. For both the etching reagents, etching was sharply interrupted by successively submerging the photographic plate in two baths with 100% isopropyl alcohol followed by drying with an air jet. The time of etching in water was 10 s, that in isopropanol solution of the glacial acetic acid was 8 s.

The efficiency of the obtained holographic gratings was estimated by measuring the grating DE as a ratio between intensity of the He$-$Ne laser beam ($\lambda = 0.63\,\mu\mathrm{m}$) diffracted in the order $+1$ and intensity of the scanning beam falling on the hologram at the Bragg angle equaling in air 28°. The scanning beam polarization was not controlled. Figs. 1, a, b present the DE dependences on the time ($t_{\mathrm{He-Cd}}$) of DCG exposure to the He$-$Cd laser radiation ($\lambda = 0.44\,\mu\mathrm{m}$) prior to and after the photographic plates processing consisting in UV-irradiation and etching. After UV irradiation, considerable DE increase (above 50$-$60%) was observed for both etching reagents. Processing of the photographic plates with UV radiation is a key moment,

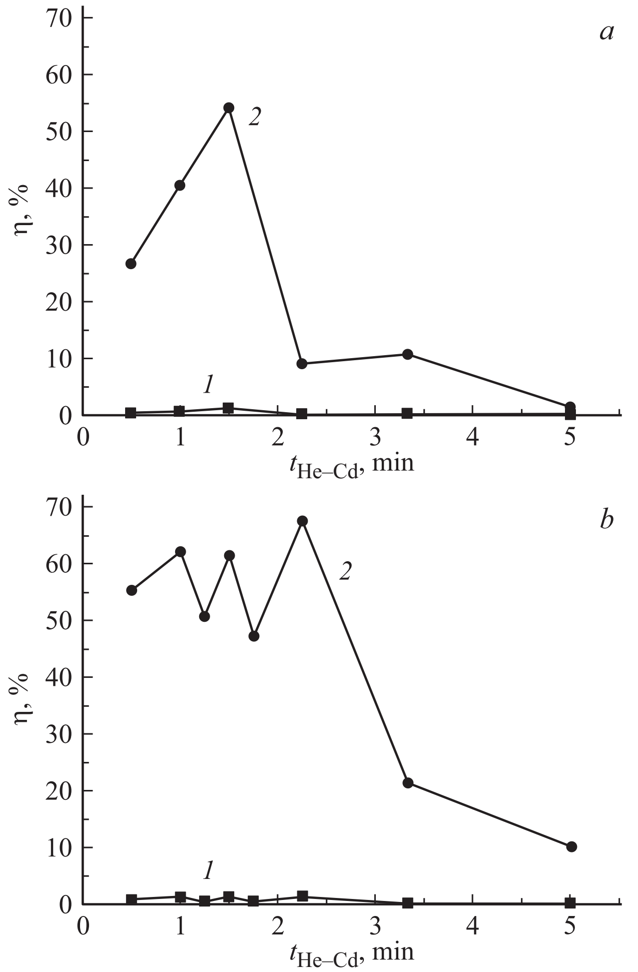

Figure 1. Diffraction efficiency η versus time $t_{\mathrm{He-Cd}}$ of grating exposure to the He−Cd laser beam. *a* — H_2O as an etching reagent, *b* — 50% isopropanol solution of CH_3COOH as an etching reagent . *1* — prior to processing, *2* — after processing.

since its exclusion from the process reduces the maximal DE of the relief-phase gratings to 3−4% independently of the etching reagent type, which was validated experimentally.

The relief-phase character of the processed holographic structures is confirmed by the angular selectivity curves. Fig. 2 presents the normalized DE versus the Bragg misalignment angle $\delta\theta_{Br}$. Experimental dots *1* represent the DE values for the gratings not exposed to UV-radiation and etching. In this case, DE does not exceed 1.2%. The obtained high angular selectivity evidences the relief-phase character of the gratings. The dashed line almost fully coinciding with the experimental dependence represents the calculations according to the Kogelnik's theory for the relief-phase grating with the thickness of 26 μm, average DCG refraction index $n_0 = 1.53$, and refraction index modulation depth $n_1 = 0.005$. The PFG-04 layer thickness measured with profile meter PROFI-130 (OJSC „Proton-MIET plant", Moscow) was 31 μm, while the Quality Certificate for the given batch of PFG-04 plates (JSC „Slavich Company", Pereslavl-Zalessky) indicates the value of $25 \pm 2\,\mu$m. All the above-specified layer thickness values are close to each other. This shows that the refraction index inner grating occupies almost the entire thickness of photographic material PFG-04.

Dependence *2* in Fig. 2 relates to DE measurements for the grating after processing consisting in UV-irradiation and etching with the 50% isopropanol solution of glacial acetic acid. The maximal DE was 65%. One can see that the Bragg's character of the angular dependence disappears, and

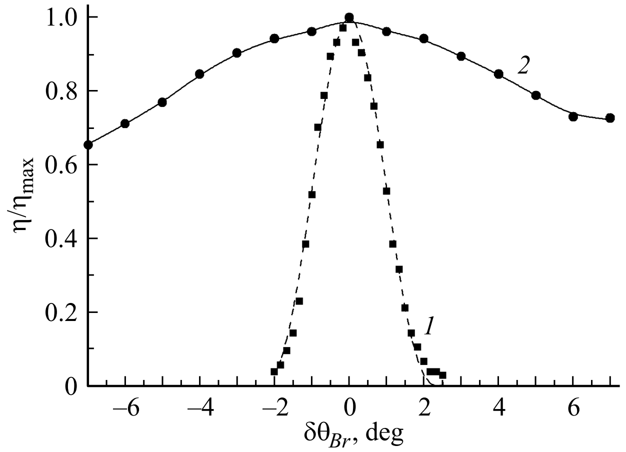

Figure 2. Experimental DE angular dependences for photographic plates PFG-04 (dots) and DE calculated in the scope of the Kogelnik's scalar theory. Experiment: *1* — prior to UV irradiation and etching, *2* — after processing consisting in UV irradiation and etching

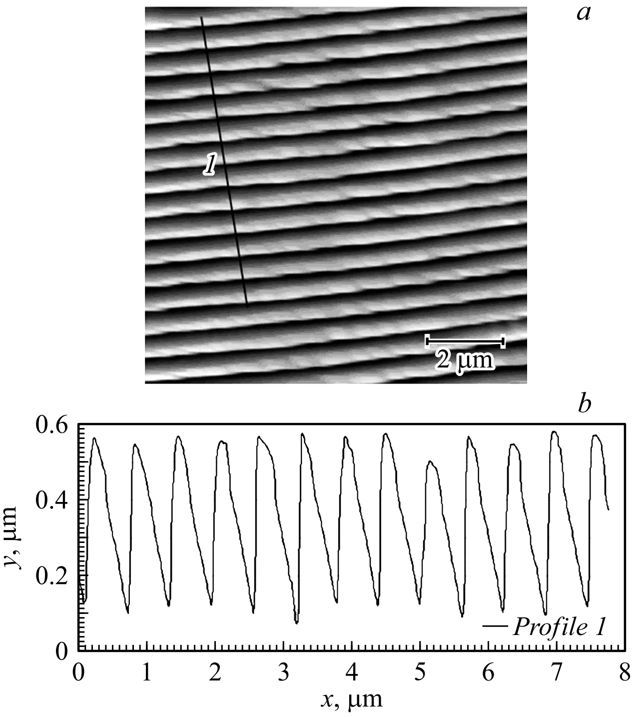

Figure 3. The image of the holographic grating surface (*a*) and a profilogram of the surface section on material PFG-04 (*b*) PFG-04 (*b*).

the refraction index relief grating transforms after processing into a surface relief grating.

In addition to the previous studies, a direct analysis of the surface structure was carried out by the atomic force microscopy. Figs. 3, *a* and *b* present, respectively, examples of the surface pattern and holographic grating relief profile obtained using an atomic force microscope Nano DST (Pacific Nanotechnology Inc.). The data are given for a grating with DE of 58% which was exposed to the He−Cd laser radiation for 3 min 20 s and UV-radiation (24 min), and then processed with the 50% isopropanol solution of glacial acetic acid.

The sample was scanned in the horizontal direction at a small angle to the grating ruling in order to reduce the material layer deformation by the probe, since the atomic force microscope worked in the contact mode using a medium-rigidity probe CSG10/W2C (produced by NT-MDT) with the typical force constant of 0.11 N/m. Average height of the relief profile (Fig. 3, *b*) in the *1* direction (Fig. 3, *a*) was $h = 0.48\,\mu$m. To acquire the true relief height, this value should be multiplied by the experimentally obtained factor of 1.3−1.8 that allows for the effect of the material deformation with the probe during scanning. Finally, the surface relief depth h was at least $0.6\,\mu$m, i.e., complied with the wavelength of the He−Ne laser scanning beam, which, according to the strict electromagnetic theory of transmission gratings [7], is necessary to achieve high DE.

Thus, this work has shown for the first time the possibility of obtaining high-frequency relief-phase gratings based on commercially produced photosensitive material PFG-04. The method used for processing the DCG layers is alternative to the conventional method for obtaining relief-phase holograms on this photographic material. The results of this study are expected to expand the application field of the holographic photographic material PFG-04 based on dichromed gelatin.

Conflict of interests

The authors declare that they have no conflict of interests.

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