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Registration of high-frequency relief-phase structures on silver halide photographic material PFG-01

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A new variant of manufacturing high-frequency holographic gratings with a spatial frequency up to $1200\,\mathrm{mm}^{-1}$ on silver halide photographic material for holography PFG-01 is proposed, the processing technology of which is tanning bleaching in bleach containing dichromates and etching in water or in solutions of glacial acetic acid in isopropyl alcohol. The key point is the destructive effect on gelatin of short-wave UV radiation with a wavelength of less than 270 nm. During the experiments, relief-phase holographic gratings with high diffraction efficiency up to a value of 42% were obtained.

Keywords: holographic diffraction gratings, silver halide photoemulsions, PFG-01 photographic plates, short-wave UV radiation, surface relief, diffraction efficiency, glacial acetic acid, isopropyl alcohol.

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Introduction

The combination of selective light tanning ($\lambda = 440\,\mathrm{nm}$) and photodegradation of gelatin by exposure to short-wavelength UV radiation ($\lambda \leq 250-270\,\mathrm{nm}$) gives good results in the manufacture of highly efficient relief-phase transmissive holographic gratings with a high spatial frequency up to $1600\,\mathrm{mm}^{-1}$ on layers of dichromated gelatin (DCG) [1–3]. It is interesting and practically useful to apply the obtained results to gelatin-containing silver halide emulsions, the sensitivity of which to light radiation exceeds the sensitivity of DCG by almost three orders of magnitude.

The concept of relief formation for DCG is that the variable tanning of the layer, created as a result of the primary registration of the interference pattern in visible light, modulates the effect of short-wave UV radiation on gelatin [4]. There is no selective tanning for silver halide emulsions, and the main factor modulating the destructive effect of ultraviolet radiation on gelatin is the presence of a silver image (SI) of the interference pattern [5]. For DCG layers, the main operation that allows to obtain a sufficiently deep relief at spatial frequencies over $1000\,\mathrm{mm}^{-1}$, is a short (10 s) etching of the layer with water or other reagents, interrupted by baths of 100% isopropyl alcohol (IPA) immediately after irradiation of the dry layer with UV radiation. In contrast to DCG, after irradiation with UV radiation, silver halide photographic emulsions require relatively long water processing procedures associated with the removal of primary SI from the entire thickness of the photographic emulsion layer [5]. This narrows the range of recorded spatial frequencies to $200-300\,\mathrm{mm}^{-1}$, which is due to the manifestation of surface tension forces that smooth the surface relief during drying of the wet colloid [6].

Based on the above differences in the properties of silver halide photographic emulsions and DCG, we can formulate requirements for changing the processing procedure described in [5], which allows to significantly expand the range of registered spatial frequencies of relief-phase holographic structures on silver halide photographic materials:

1. Selective tanning of photographic emulsion (structuring) can be carried out chemically using tanning bleaches such as R-10 [7] and R-9 [8] and combining this operation with the removal of silver compounds from the emulsion layer.

2. Ultraviolet irradiation of the dried photographic emulsion is carried out after the removal of silver compounds from the entire thickness of the layer.

3. Short etching of the photographic emulsion that acts only on the near-surface layer, is carried out immediately after exposure to short-wave UV radiation.

The table allows one to compare in detail the procedures for processing DCG layers and silver halide photographic emulsions, as a result of which relief-phase holographic structures are obtained on gelatin, a material with relatively high optical parameters.

Experimental technique and research results

In this study, silver halide photographic material PFG-01 for holography with a thickness of $6\,\mu\mathrm{m}$ (manufactured by LLC „Company Slavich“, Pereslavl-Zalessky) was chosen.

Comparison of the treatment of DCG layers and silver halide emulsion

DCG	SILVER HALIDE PHOTOGRAPHIC EMULSION
Exposure to coherent light from of a He–Cd laser ($\lambda = 440$ nm). Initiation of the process of selective light tanning in accordance with the interference pattern of the hologram.	**Exposure to coherent light of a He–Ne laser. ($\lambda = 630$ nm).** Latent image formation.
Water treatment in sodium sulfite solution. Removal of coloring chromium compounds from the layer.	**Photographic development in a contrast developer.** Formation of the SI interference pattern of a hologram.
Washing. Completion of the gelatin selective tanning process.	**Washing**
Drying	**Fixing.** Silver halide removal.
	Drying.
	Rehalogenating tanning bleaching in the solutions containing dichromates. Selective chemical tanning of gelatin.
	Washing.
	Fixing. Silver halide removal.
	Washing
	Water treatment in sodium sulfite solution. Removal of coloring chromium compounds from the layer.
	Washing
	Drying.
UV irradiation of the layer. Selective photodegradation of gelatin.	**UV irradiation of the layer.** Selective photodegradation of gelatin.
Short-time etching. Relief formation on the gelatin surface.	**Short-time etching.** Relief formation on the gelatin surface.
Isopropanol baths. Interruption of the etching process.	**Isopropanol baths.** Interruption of the etching process.
Final drying.	**Final drying.**

Holographic gratings were recorded by He–Ne laser radiation with a wavelength of 630 nm in a symmetric optical scheme. The grating frequency was about 1200 mm^{-1}. A DRT-220 high-pressure mercury lamp served as a source of short-wave UV radiation with a wavelength of less than 270 nm.

Three series of experiments were carried out to create high-frequency holographic gratings, differing both at the bleaching stage (tanning direct or reversal bleaching) and the gelatin etchant option (water or a solution of glacial acetic acid (GAA) in IPA). The exposed grating samples were chemically processed in a contrast developer D-19 for 3 min and fixed in an acid fixer for 10 min, which led to the creation of SI in the emulsion layer. Then the samples were washed and dried on air.

In the first series of experiments, direct bleaching in R-10 bleach, fixing to remove SI from the emulsion, washing, brightening in a sodium sulfite solution to remove coloring Cr compounds from the emulsion layer, washing and drying were carried out. At this stage, the DE of primary holograms was measured as the ratio of the energy of light diffracted into the first order of diffraction to the energy of the beam incident on the sample at an optimal angle of incidence close to the Bragg angle (Fig. 1, curve *1*). This was followed by irradiation of the samples with a mercury-quartz lamp for 30 min, etching in the solution GAA (50%) + IPA (50%) for 15 s, and washing in two baths IPA (100%), 30 s each. After final drying with air blowing, the DE of the gratings was measured. Curve *1* in Fig. 2 shows these measurements. The maximum DE was about 42%.

In the second series of experiments, after irradiation of the samples with UV radiation, etching in water for 12 s

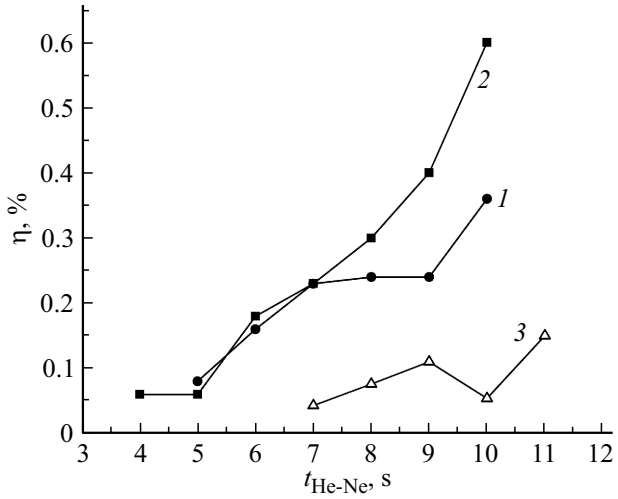

Figure 1. Dependence of the DE of η of primary holographic gratings on the time of exposure to He−Ne laser radiation for three options for processing a silver halide emulsion before UV exposure and etching: *1, 2* — direct rehalogenating bleaching, *3* — reversal whitening.

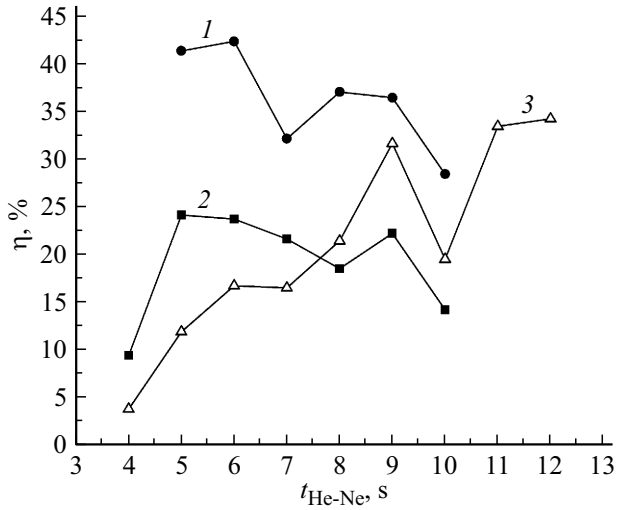

Figure 2. Dependence of DE of relief-phase holographic gratings on the time of exposure to He−Ne laser radiation for three options for photoemulsion processing after exposure to UV radiation and etching: *1, 3* — etching in the GAA solution (50%) + IPA (50%), *2* — etching in water.

was used instead of etching the layer in GAA (50%) + IPA (50%). The dependence of the gratings DE in this case is represented by curves *2* in Figs 1 and 2, respectively (before and after UV exposure and etching, respectively).

The third series of experiments was carried out using R-9 reversal bleach and etching in GAA (50%) + IPS (50%). In this case, the need for a second fixation disappeared, because SI was removed from the photoemulsion layer directly during reversal bleaching. The gratings DE for this case is represented by curves *3* in Figs 1 and 2 (before and after UV exposure and etching, respectively). The maximum DE reached 34%.

Conclusions

The technology for processing photographic material for holography PFG-01 based on the destructive effect of UV radiation on gelatin, including the operations of tanning bleaching and short etching, allowed us to expand the spatial range of relief-phase holographic structures recorded on silver halide emulsion, up to 1200 mm^{-1}. The DE of primary holographic gratings, measured before samples were irradiated with short-wavelength UV radiation, increases as a result of the proposed processing by a factor of 200−500, reaching a maximum value of 42% for the obtained relief-phase structures.

Conflict of interest

The authors declare that they have no conflict of interest.

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