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SILVER-HALIDE GELATIN HOLOGRAMS.(U)
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Silver-halide gelatin holograms

B.J. Chang and K. Winick

Radar and Optics Division
Environmental Research Institute of Michigan
P.O. Box 8618, Ann Arbor, MI 48107

Abstract

Volume phase holograms, whose holographic characteristics are similar to those of conventional dichromated gelatin holograms, can be formed employing commercial silver-halide films. Major advantages of silver-halide gelatin holograms are wide spectral response and lower exposure requirement.

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Introduction

The dichromated gelatin process can produce nearly ideal holograms which have high diffraction efficiency and little scattering and absorption [1-3]. However, dichromated gelatin has two basic drawbacks. The first is the limited spectral response; dichromated gelatin is primarily sensitive to ultraviolet through blue-green radiation. The second is the relatively high exposure requirement; a typical exposure at 514.5 nm is about 50 to 100 mj/cm^2. Although dichromated gelatin can be made red-sensitive with proper dye-sensitization [4, 5], the exposure of red-sensitized dichromated gelatin is considerably high: i.e., 200 to 1000 mj/cm^2 at 632.8 nm.

Since the proposed silver-halide gelatin uses silver-halide (or other silver-salts including dye-sensitized silver-salts) as a sensitizer, its spectral and exposure sensitivities are equivalent to those of conventional holographic films. The main difference between the dichromated gelatin process and silver-halide process is the creation of a hologram latent image which is given in the form of hardness differential between exposed regions and unexposed regions. The latent image of a dichromated gelatin hologram is created by the photochemical hardening reaction mechanism between dichromates and gelatin during the exposure [2, 3, 6]. On the other hand, the latent image of a silver-halide gelatin hologram is formed by the reaction products of either tanning development or tanning bleach which harden the gelatin by forming cross-link bonds between long gelatin molecules [7].

In the following sections, we will describe a method for forming silver-halide gelatin holograms and show our initial experimental results obtained by employing Kodak 649F plates and a He-Ne laser.

Basic processing principles

Both dichromated gelatin and silver-halide gelatin holograms are essentially almost pure gelatin holograms. The latent images of both are given in the form of hardness differential between the exposed and unexposed regions. The latent image of the dichromated gelatin hologram is created by the photochemical reaction during the exposure while the latent image of the silver-halide gelatin hologram is created by the reaction products of conventional photographic development and bleach process. Except for the creation of latent images, both dichromated gelatin and silver-halide gelatin hologram processes are basically identical.

First let us review the latent image formation of dichromated gelatin. When a dichromated gelatin film is exposed to actinic radiation, the hexavalent chromium ion Cr^{6+} is either directly or indirectly photo-induced to trivalent chromium ion Cr^{3+} which is thought to form a cross-link bond between neighboring gelatin strands [6]. This cross-link bond hardens the gelatin creating a hardness differential between highly exposed regions and relatively unexposed regions.

In the case of silver-halide gelatin holograms, the reaction products generated during either tanning development or tanning bleach harden (or tan) the gelatin by forming cross-link bonds between long gelatin molecules. Since the reaction products are formed in the immediate vicinity of the reduced silver, the exposed regions become harder than the unexposed regions. Once the latent hologram image is created as a form of hardness differential, the developed silver grains and silver salts are removed before the isopropanol development (or dehydration).

The refractive index modulation for both cases is achieved by converting a hardness differential (i.e., latent hologram image) to a gelatin density differential [3, 10] during the final alcohol development steps.

For our present silver-halide gelatin hologram process, we have employed modified R-10 type bleaches [8]. Table I shows formulas for stock solutions and the procedure for preparing a bleach solution. During the bleaching process, the metallic silver may be oxidized to Ag^+. In the immediate vicinity of the oxidized silver grains, trivalent chromium ions are generated which form cross-link bonds between neighboring gelatin molecule strands. Cross-link bonds harden exposed gelatin regions.

The probable chemical oxidation reaction [11] is

$$Cr_2O_7^{2-} + 14H^+ + 6Ag^o \rightarrow 2Cr^{3+} + 7H_2O + 6Ag^+.$$

The oxidized silver ion Ag^+ eventually forms such silver-salt compounds as AgCl, AgBr, and AgI which are removed during the following fixing bath. The life span of the chromium ion Cr^{3+} can be controlled by adjusting the ratio of stock solution B to stock solution A. As the content of solution B increases, the life span of the chromium ion decreases and the cross-link bonds occur near the metallic silver because the chromium ion Cr^{3+} quickly forms such chromium salts as $CrCl_3$, CrI_3 and $CrBr_3$. However, as the content of solution B decreases, the life span of chromium ion increases and Cr^{3+} ions diffuse into the unexposed regions to form cross-link bonds, and thus may diminish the latent image contrast. On the other hand, a large quantity of solution B produces a significant amount of chromium salts to produce visible dark stain on the exposed regions.

Properties of silver-halide gelatin hologram gratings

In order to investigate the holographic properties of the proposed silver-halide gelatin holograms, we recorded hologram gratings in Kodak 649F plates at 632.8 nm. Our present processing procedures are shown in Table II. We used the R-10 bromide bleaching for creating the hologram latent image. Since we used the Kodak D-19 non-tanning developer, the primary source of hardening is the chromium ion Cr^{3+} reaction product of the ammomium dichromate in the R-10 potassium bromide bleach solution. Similar results were obtained with either R-10 type bleach solutions, such as R-10 potassium iodide and R-10 sodium chloride, or Kodak R-9 [9].

Figure 1 shows the diffraction efficiency as a function of exposure. Since the Kodak 649F plate has a broad spectral range, similar diffraction efficiency curves can be obtained with holograms recorded at either 488 nm or 514.5 nm. The diffraction efficiency is defined as the ratio of the intensity of the 1st order diffraction, I_1, to the incident light intensity I_i and the average transmittance, T_a, is defined as $\sum_{j=1}^{N} (I_1 + I_0)_j / NI_i$ where I_0 is the intensity of the 0-th order diffraction and N is the total number of holograms recorded in the same plate.

The average transmittance of holograms shown in Figure 1 was about 75%. Assuming that the front and rear surface reflection loss was about 7%, we can easily find that the average absorption was about 18%. This absorption was primarily due to the stain caused by the reaction products such as $CrBr_3$.

In order to reduce the absorption caused by the stain, we have reduced the silver-halide grain population by soaking unexposed plates in a properly diluted fixer solution without hardener. The reduction of silver-halide grain population decreases the absorption coefficient of plates and makes exposure rather uniform as a function of hologram depth. The increased exposure uniformity through the hologram depth may also increase the overall refractive index modulation. Since the absorption constant of the original Kodak 649F plates is relatively high, i.e., more than 50%, the light intensity rapidly decreases as it propagates through the medium and thus the front surface of emulsion is usually overexposed while the rear surface of emulsion is underexposed. This high absorption coefficient could reduce the overall refractive index modulation and also increase the chromium salt population which reduces the transmittance of holograms.

Using the preprocessing procedures shown in Table III, we preprocessed Kodak 649F plates and made exposure tests. As shown in Figure 2, the diffraction efficiency as well as the average transmittance of holograms varies according to the preprocessing condition. The holograms recorded in a preprocessed plate with a 10% solution of Hunt standard fixer

(without hardener) for holographic plates show the highest diffraction efficiency and the lowest absorption. The holographic characteristics of silver-halide gelatin holograms appeared to be comparable to those of dichromated gelatin holograms.

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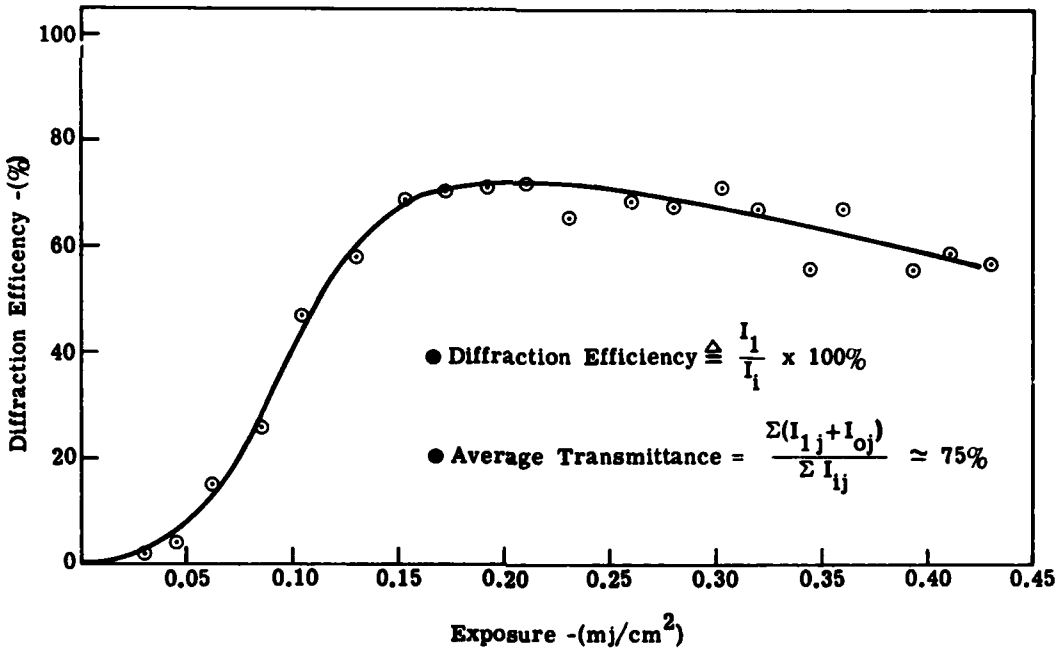

Figure 1. Diffraction efficiency as a function of exposure for silver-halide holograms recorded in Kodak 649F plates.

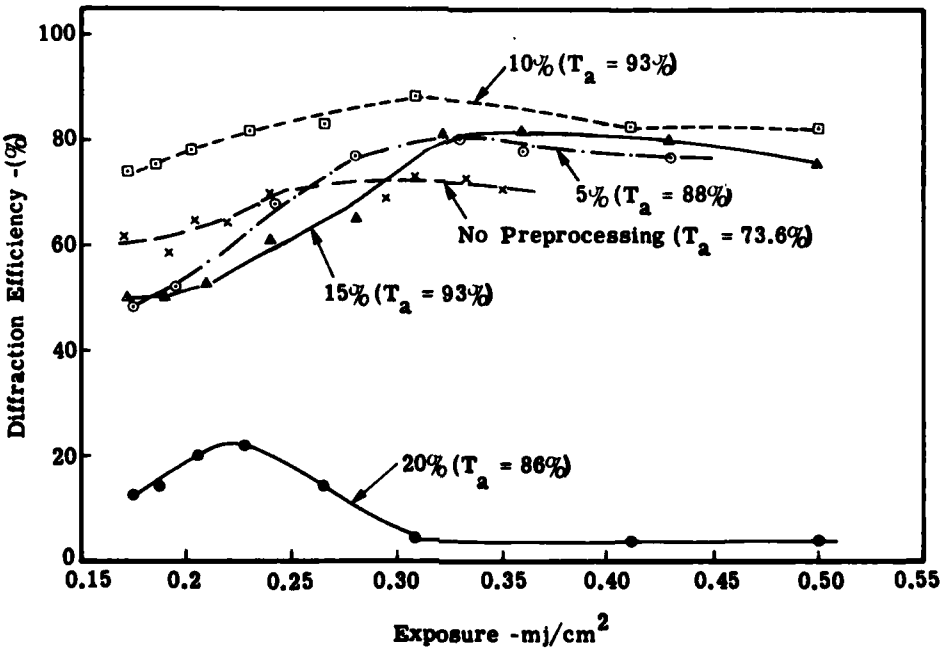

Figure 2. Effects of preprocessing on the diffraction efficiency and *average transmittance*.

TABLE I
PREPARATION OF R-10 TYPE BLEACHES

Stock Solution A:

Distilled water - 500 ml
Ammonium dichromate - 20g
Concentrated sulfuric acid - 14 ml
Distilled water to make - 1000 ml

Stock Solution B:

Potassium bromide - 92g (or potassium Iodide - 128g, or Sodium Chloride - 45g)
Distilled water to make - 1000 ml

Mixing Procedure of Bleaching Solution

Just before use, mix one part A with ten parts distilled water and then mix one part B with the diluted part A solution.

TABLE II
PROCESSING PROCEDURES OF SILVER-HALIDE GELATIN HOLOGRAMS.
(All Steps, except where noted, are at a temperature of 72°F)

Step	Directions	Lightening
1	Develop with Kodak developer D-19 for 5 minutes	Dark
2	Rinse in stop bath for 30 seconds	Dark
3	Rinse in running water for 15 seconds	Dark
4	Bleach in a modified R-10 solution for 1 minute after the plate has cleared (plate becomes usually clear in 90 seconds) - see Table I for the preparation of a bleach solution	Red Light
5	Rinse in running water for 15 seconds	Red Light
6	Soak in 0.5% ammonium dichromate for 5 minutes	Red Light
7	Soak in Hunt standard fixer with 0 to 2%* (in volume) hardener for 5 minutes	Red Light
8	Wash in running water for 10 minutes and rinse in distilled water for 3 minutes	Room light
9	Dehydrate (or develop) in 50/50 solution of distilled water and isopropanol for 3 minutes	Room light
10	Dehydrate in 100% isopropanol for 3 minutes	Room light
11	Dry in either air or vacuum chamber	Room light

*Fixer with x% hardener

$$x\% = \frac{\text{hardener volume}}{\text{water volume} + \text{concentrate volume}} \times 100\%$$

TABLE III
PREPROCESSING PROCEDURES
(All steps, except where noted, are at a temperature of 72°F and are in dark)

Step	Directions
1	Soak in a x%* solution of Hunt standard fixer for plates without hardener for 3 minutes
2	Wash in running water for 10 minutes
3	Soak in distilled water for 2 minutes
4	Dry in air overnight

*x% of Hunt standard fixer for plates (HSFP) without hardener

$$x\% = \frac{\text{HSFP volume}}{\text{water volume + HSFP volume}}$$