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A COOKBOOK FOR DICHROMATED GELATIN HOLOGRAMS,(U)
JAN 79 D GUENTHER, C D LEONARD

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A COOKBOOK FOR DICHROMATED GELATIN HOLOGRAMS

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Research Directorate
Technology Laboratory

12 January 1979

U.S. ARMY MISSILE RESEARCH AND DEVELOPMENT COMMAND

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Gelatin sensitized by ammonium dichromate (DGH) produces a near-optimum medium for holography. Holograms produced in this medium have very low scattering noise and very high diffraction efficiencies. This report summarizes techniques found to be useful in producing high quality holograms.

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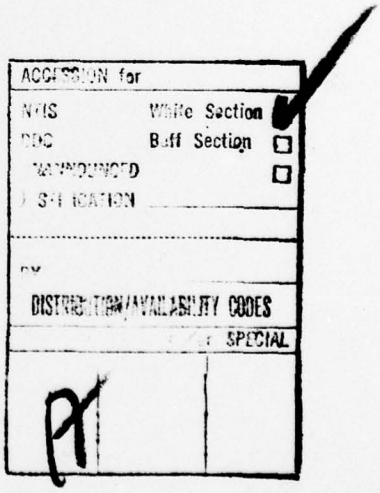

I. INTRODUCTION

Gelatin sensitized by ammonium dichromate (DGH) produces a near-optimum medium for holography. Holograms produced in this medium have very low scattering noise and very high diffraction efficiencies. This report summarizes techniques found to be useful in producing high quality holograms.

The processing described is largely based on the procedure of Colburn and Chang [1] developed at the Environmental Research Institute of Michigan. Credit should be given to Zech [2] of Radiation, Inc., for the generalized procedure of incorporating the preprocessing techniques of Chang [3] and the postprocessing techniques of Lin [4]. Their procedures have been modified to reduce the overall processing time.

There is still a great deal that is not known about the chemistry of dichromated gelatin. The procedures outlined in this report will result in high quality holograms, but none of the procedures may be optimal. A more detailed evaluation of DGH has been described [5].

Five areas will be covered: general preparation, plate preparation, exposure, development, and assembly. An outline of the procedure is given in the Appendix.

II. GENERAL PREPARATION

For processing of DGH, it is imperative that the relative humidity of the laboratory be kept under 40% from gelatin sensitization to final assembly of the hologram. The relative humidity can vary markedly even within a single room; therefore, it is recommended that several hydrometer gauges be used to monitor the laboratory environment. It should be noted that hydrometers, even as received from the manufacturer, can be out of calibration. It is recommended that a wet-dry bulb thermometer be used for periodic calibration. Several small room dehumidifiers will usually reduce the relative humidity below 40%. Distilled water is used in making all solutions; washes were done using temperature controlled tap water.

III. PLATE PREPARATION

For convenience, it is usually desirable to initially purchase commercially available photographic films or plates. The silver halides and dyes can be removed and the remaining gelatin sensitized with ammonium dichromate. The controls used by the film manufacturer in the production of silver halide holographic films and plates do not appear to be coincident with the controls needed to produce good DGH. Thus, good holographic films may not be good DGH films. We have had satisfactory results using Kodak 649F and 131 emulsions. The performance of these emulsions will

vary according to batch and/or age. Exposure and processing tests should be performed before every critical exposure, at least until experience is gained in working with DGH.

Having selected one or more emulsions for testing, the plates are preprocessed and sensitized according to the following procedure:

a) Preprocessing - For as many plates as can be simultaneously processed:

1) P1 - Fix 10 minutes in Kodak Rapid Fixer without hardener.

2) P2*- Wash 5 minutes at 90°F, initially raising(and then lowering) the temperature gradually (5 to 10 min.) from room temperature (~70°F).

3) P3*- Wash 5 minutes in Kodak Rapid Fixer with hardener.

4) P4 - Wash 10 to 12 minutes at 70°F, running water.

5) P5 - Rinse 1 minute in Photoflo (one drop Kodak Photoflo 200 in 500 ml of water).

6) P6 - Dry by wiping with wiper blade.

b) Sensitizing - For plates to be used within 8 hours:

1) S1 - Sensitize 5 minutes in X% solution of ammonium dichromate (X gm of ammonium dichromate in 100 ml of water with 0.X ml of Photoflo).

2) S2 - Wipe with wiper blade.

3) S3 - Dry on hotplate for 3 minutes at 160°F.

4) S4 - Allow to cool to room temperature for 1 hour.

The concentration of ammonium dichromate used in the sensitization of the gelatin controls the exposure time and the Bragg angle shift. The amount of hardener used in the fixer for both preprocessing and development controls the diffraction efficiency and scattering noise. These two facts again point out the need for making test exposures.

Two methods can be used for drying the sensitized plates. Softer emulsions may be dried in a horizontal position at room temperature. A second, faster technique useful with harder emulsions is to wipe the surface of the gelatin with a wiper blade (windshield wiper blades

*Kodak 649F emulsion only

designed for older model VW's with flat windshields are excellent). After wiping, the plate is placed on a preheated hotplate (~160°F - you should be able to touch the hotplate but not leave your finger on it). The actual temperature of the hotplate is not very important; we use one that drys the plates in approximately 3 minutes. If crystalization occurs on the gelatin surface beyond a narrow edge region, then the plate should be placed into the sensitizing solution again. A high room temperature or a high substrate temperature can result in rapid crystalization before the wiping process is complete (this would occur when using very high concentrations) and can be solved by simply lowering the temperature of the plates. After drying, the plates should appear clean with no streaks, smudges, or crystals.

The rapid drying procedure appears to affect the dichromate concentration required for a given holographic performance. When the wiping process is used, three times the dichromate concentration used in the nonwiping process is needed to produce a similar effect. For example, to obtain no Bragg angle shift, holograms made using the nonwiping technique required concentrations of 6 to 8%. When the wiping technique was used, concentrations of 20 to 25% were required with the lower concentration corresponding to the requirements of reflection holograms while the higher values correspond to transmission holograms.

The sensitizing solution and the sensitized plates should be exposed only to red light (for example, Kodak Safelight Filter 1A). The color of the plates and solution changes from a yellow to a brown with exposure to light or through ageing. Low concentrations can be stored by refrigeration. At high concentrations, the solution lasts approximately 8 hours and the plates approximately 4 hours. The plates can usually be washed and reused if some darkening has occurred. The reader should note that the hardness of the gelatin is modified as the plates darken.

IV. EXPOSURE

Once the plates have been sensitized, the relative humidity should be kept below 40%. It has been found that the sensitized emulsion can be covered with a glass plate using xylene as a liquid gate. By doing this, the relative humidity of the exposure laboratory need not be held below 40%. The gate can drain and evaporate during long exposures. Operating times of over an hour have been obtained by sealing the gate with aluminum tape (the type used for making lantern slides). Exposure times can range from 50 to 1000 mJ/cm^2 at 5145 $\AA$ depending on the dichromate concentration and the percent modulation required. A good starting point would be an exposure of 250 mJ/cm^2 at a dichromate concentration of 5%.

V. DEVELOPMENT

For processing of plates immediately after exposure:

a) D1 - Agitate 5 minutes in 0.5% ammonium dichromate solution (can be made from the sensitizing solution).

b) D2*- Agitate 5 minutes in Rapid fixer without hardener (with hardener for 649F).

c) D3 - Wash 10 minutes in running water (70°F for 649F and 90°F for other emulsions).

d) D4*- Agitate 3 minutes in 50:50 solution of isopropyl alcohol and water; then wipe off back and edges.

e) D5*- Vigorously agitate 3 minutes in 100% isopropyl alcohol.

f) D6 - Dry standing on edge (649F), or wipe with blade and heat on hotplate.

Except for the 50:50 solution of isopropyl alcohol in water, all solutions should be discarded after one use. To reduce the quantity of chemicals used, tray development is used with only enough liquid to barely cover the plates.

The gelatin emulsions have different degrees of hardness depending on their age and on the manufacturing process. For example, the gelatin on thin plates has been found to be harder than the same emulsion type on microflats. It is useful to have stock fixer solutions with and without hardener to allow the correct percentage of hardener to be selected for Step D2 during the initial test exposures. This type of processing control need be applied only for critical control of the DGH performance.

The most important developing step is the 100% isopropyl alcohol bath. In this step, rapid and complete dehydration of the gelatin is required. Excess liquid is wiped from the back and edges of the plate (not the emulsion). The plate is placed, emulsion side up, in a tray and the alcohol is poured directly onto the plate. Rapid agitation should begin immediately and should continue for the entire 3-minute wash. The agitation cannot be too rapid. The 100% isopropyl alcohol can be retained for later use in making up 50% alcohol/water solutions.

Drying may be accomplished in two ways. Softer emulsions may be placed on end to drain and dry. The results using this drying process

*Venting of the darkroom may be required for these steps.

will be variable. Absolute control of the relative humidity is required for consistent results.

The second drying technique involves first removing the excess liquid from the emulsion with a wiper blade and drying on a hotplate. The hotplate should provide uniform heating of the DGH. If processing nonuniformities are observed after drying, the plate may be returned to the 50:50 alcohol/water solution and reprocessed.

After 1 minute at Step D2, room lights may be used. Steps D3 through D6 may be repeated if the processed plates are accidentally exposed to moisture. These steps may also be repeated with the wash temperature at D3 increased. This reprocessing can often increase the diffraction efficiency to recover what would normally be an unusable hologram [6]. There does not appear to be a limit to the number of times these final processing steps may be repeated.

VI. ASSEMBLY

Moisture will degrade the holograms. To make DGH rugged, a cover plate is cemented over the gelatin. The same type of protection is obtained when multiple holograms are assembled emulsion-to-emulsion. Before assembly, the plates are baked at 160°F in a vacuum for approximately 2 hours to remove water from the gelatin. American Handicraft Company's "Clear Cast" liquid casting plastic or Summers "Lens Bond" optical cement available from Edmund Scientific Company were used as the cement. Five cubic centimeters of the cement is mixed with one drop of catalyst and placed in a vacuum of 25 to 26 in. until all of the bubbles were removed. If too high a vacuum is used, the solvent evaporates making the cement unusable. After the bubbles have been removed from the cement, it is poured over the gelatin surface of the hologram in an "X". The cover plate or the gelatin surface of another hologram is then slowly rolled onto the "X" allowing the cement "X" to spread to the plate edges. Care should be taken that no air is trapped as the "X" spreads. The sandwich of glass and cement is then heated on a hotplate at 140° to 160°F until the plastic becomes firm but tacky. A razor blade can then be used to trim away excess from the edges of the plates, and acetone can be used to remove the cement from the plate faces.

The following is a listing of the previously described steps of the assembly procedure:

a) A1 - Bake holograms at 160°F for 2 hours under vacuum.

b) A2 - Mix 5 cc's of cement and one drop catalyst.

c) A3 - Place cement in vacuum to remove air bubbles.

d) A4 - Cement glass plates together.

e) A5 - Bake cemented plate at 140° to 160°F until cement is tacky.

If the need arises, the plates can later be separated by dissolving the cement in acetone. The holograms are not damaged by exposure to acetone, but must be reprocessed starting at Step D3.

The most important step in this final assembly is Step A1, the vacuum bake-out. If this step is carried out, the covered hologram will survive exposure to boiling water.

A dry box is a convenient accessory for storing processed plates until one is able to cover the gelatin with a protective plate.

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Appendix. PROCESSING TECHNIQUES

The procedures used here are derived from those of B. J. Chang. One procedure is used for 649F emulsions and a second is used for all other emulsions. Exposure and processing are done in rooms where the relative humidity is held below 40%. Unless otherwise noted, all washes are temperature controlled and last for 10 minutes. All plates are dried by first wiping off all excess liquid with a rubber wiper blade and then drying them in a horizontal position on a hotplate set at 160°F. A thin photographic plate is removed after 3 minutes and a thick (0.25 in.) photographic plate is removed after 4 minutes.

Preprocessing

For 649F plates, the following procedure is used:

1) P1 - Fix 10 minutes in Kodak Rapid Fixer without hardener.

2) P2 - Wash 10 minutes in 90°F water.

3) P3 - Wash minutes in Kodak Rapid Fixer with hardener.

4) P4 - Wash 10 minutes.

5) P5 - Rinse 1 minute in Photoflo.

6) P6 - Wipe with wiper blade.

All other plates are preprocessed without Steps P2 and P3.

Sensitizing

The following procedure is the same for all emulsions. It is conducted in red light (Kodak Safelight Filter 1A):

1) S1 - 5 minutes in X% solution of ammonium dichromate with 0.X% of Kodak Photoflo.

2) S2 - Wipe with wiper blade.

3) S3 - Dry on hotplate for 3 minutes at ~160°F.

4) S4 - Remove crystalized ammonium dichromate from the back of the plate.

5) S5 - Allow to cool for 1 hour.

At the end of the sensitizing process, the plates should appear clean with no streaks, smudges, or crystals. If crystals of ammonium dichromate form on the gelatin, the sensitizing process must be repeated.

Exposure

It is recommended that the sensitizing plates be used the same day. If the sensitizing solution were 10% or less, the plates can be stored overnight in a refrigerator provided the plates are sealed from moisture. The plates required from 50 to 1000 mJ/cm^2 at 5145 Å to produce a hologram. The actual exposure energy is determined by the percent modulation required and the concentration of ammonium dichromate used. Test exposures should be run to insure optimum performance.

Development

The following developing procedure was used with modifications required for Kodak 649F plates shown (Steps D1 and D2 are performed in red light):

1) D1 - Agitate 5 minutes in a 0.5% ammonium dichromate solution.

2) D2 - Agitate 5 minutes in fixer without hardener (use fixer with hardener for 649F).

3) D3 - Wash at 90°F (70°F for 649F).

4) D4 - Rinse 3 minutes in a 50:50 solution of distilled water and isopropyl alcohol, then wipe off back and edges of plate.

5) D5 - Rinse 3 minutes in 100% isopropyl alcohol with rapid agitation.

6) D6 - Dry (do not use a hotplate for 649F plates).

The 100% isopropyl alcohol used in Step D5 can only be used to process one plate. The index of refraction modulation can be changed by repeating Steps D3 through D6. To increase the modulation, a higher wash temperature can be used [up to 110°F (90°F for 649F)].

Assembly

The assembly procedure was as follows:

1) A1 - Vacuum bake DGH at 160°F for 1 hour.

2) A2 - Mix 5 cc cement and one drop catalyst.

3) A3 - Place cement in vacuum to remove air bubble.

4) A4 - Cement glass plates together.

5) A5 - Heat cemented plates at 140-160°F until cement is tacky.

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