

Characterization of High Resolution Photographic Emulsion BB640 by Holographic Methods

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Abstract

The gap created in the last decade in the market of fine and ultra-fine grain silver halide emulsions with the withdrawal of the historical producers was filled in recent years with new products and manufacturers. We have studied with holographic techniques the emulsion BB640, with a mean grain size of 20 nm, and we have found out that we can easily reproduce the results obtained with the discontinued materials. Additionally, we have found new features with processing techniques not used with former emulsions, obtaining densities higher than 11. We have obtained very good results with spatial frequencies higher than 7000 l/mm and diffraction efficiencies higher than 70 %. The material, in its monochromatic or panchromatic version has been used to spatially and spectrally multiplex holograms obtaining very high diffraction efficiencies.

Emulsion characteristics

Ultra-fine grain holographic emulsion BB640, manufactured by British company Colourholographics Ltd., has a nominal grain size of 20 nm [1] and its sensitivity in the red region of the spectrum, with a transmittance of the unexposed emulsion at 632.8 nm of 71.3 %. Thickness and mean refraction index of unexposed BB640 plates have been calculated from interferometric data with a thin film resonance method. Values obtained for unexposed emulsion are a thickness of 9.3 microns, a mean refraction index of 1.579 and a attenuation coefficient of 33.4 cm^{-1} .

Methodology

Prior to exposure, plates need to be presensitized to recover or even increase the original sensitivity. Several procedures were used, but the best one was a 3% Triethanolamine (TEA) water solution bath followed by a water bath. After that, plates were dried with a photographic roll and warm

air and left in the exposing room to cool down for half an hour in normal laboratory conditions.

Plates were holographically characterized for recording transmission and reflection holograms. The first was done by using the plates for recording volume unslanted holographic transmission gratings with a spatial frequency of 1200 lp/mm obtained by the interference of two symmetrically incident collimated laser beams from a p polarized He-Ne laser ($\lambda = 632.8 \text{ nm}$) at an angle of 22.3° each. The reflection study was done recording volume unslanted holographic reflection gratings with a spatial frequency of 4992 l/mm obtained with a normal incident single collimated He-Ne beam in a Denisyuk configuration. For both studies, plates were mounted on a computer controlled motorized holder which enable us to record 9 gratings with different exposure energies on a 2.5"x2.5" plate.

Exposed plates were processed with a variety of developers, fixers and bleaches. The developers used were: AAC (Ascorbic Acid 18 g/l + Sodium Carbonate 60 g/l), PAAC (AAC with 0.5 g/l of Phenidone), CAAC (Ascorbic Acid 5 g/l + Sodium Carbonate 30 g/l + Pyrocatechol 10 g/l + Sodium Sulphite 5 g/l + Urea 50 g/l), PMAAC (AAC with 0.5 g/l of Phenidone + 2 g/l of Methol), Pyrogalol (Pyrogalol 5 g/l + Sodium Sulphite 15 g/l + Sodium Carbonate 60 g/l), Kodak D19 and Kodak D8. The fixer used in the density studies was non-hardening Kodak F-24. Bleachings used were: direct rehalogenating ferricianide bleach, modified fixation-free rehalogenating bleach Kodak R-10 (Potassium Dichromate 2 g/l + Sulphuric Acid 10 cc/l + Potassium Bromide 35 g/l) and solvent bleach Kodak R-9 (Potassium Dichromate 2 g/l + Sulphuric Acid 10 cc/l). After bleaching, plates were washed and soaked in deionized water with some drops of Kodak Photoflo 200 solution and Acetic Acid to prevent printout, and dried in cited normal laboratory conditions.

Densities were calculated from transmission measurements taken with a solid state detector ranging from 20 Watts/cm^2 to 0.0001 nanoWatt/cm^2 . The probe beam was a slowly expanded laser beam with an intensity of

over 20 mW/cm^2 . With this configuration it is not possible to measure transmission values corresponding to densities higher than 11.3, so all graphics will show that limit. Transmission values of 0.0000 nW/cm^2 were measured, indicating higher densities outside the limits of our detector. Diffraction efficiency (DE) measurements of the bleached recordings were made using a collimated He-Ne laser beam at the grating Bragg angle.

Results

The set of developers and bleaches mentioned above have been used to compare the holographic characteristics of the diffraction gratings.

Developers

Developers used were compared by their measured D-LogE curves with the holographic transmission setup. Fig. 1 present the different D-Log E curves for each of the developers described above. Plates were fixed with non-hardening fixer Kodak F-24. Kodak D8 curve has been omitted since it will be discussed separately in a further section. Maxima densities were obtained for AAC and CAAC developers, with values of 7.9 ($E=1920 \mu\text{J/cm}^2$) and 8.1 ($E=3840 \mu\text{J/cm}^2$) respectively.

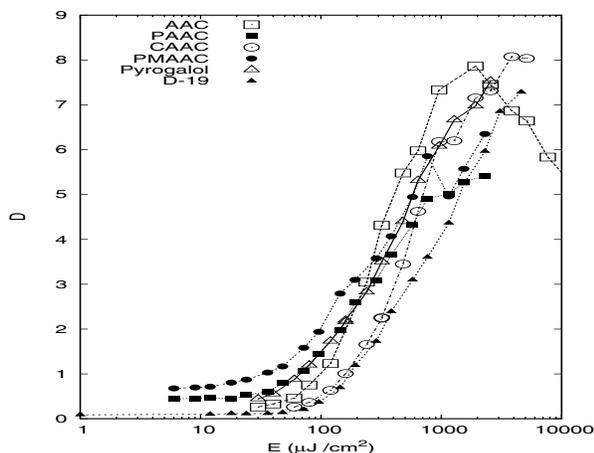


Figure 1: D-Log E curves corresponding to emulsion BB640 developed with AAC, PAAC, CAAC, PMAAC, pyrogalol and Kodak D-19 developers.

Bleaches

In order to study the holographic response of the material, the above set of developers was applied to holographic transmission gratings with the three bleaching methods described in the former section. The direct ferric

bleach was used with the fixed plates after the measurements of the densities have been performed. Fixation free rehalogenating bleach R-10 study was done with a different set of plates. Solvent (reversal) bleach will be discussed later. Figs. 2 and 3 show the results. For the ferric bleach, the highest diffraction efficiencies obtained were for developer D-19 with 73% ($768 \mu\text{J/cm}^2$), PAAC with 69.2% ($384 \mu\text{J/cm}^2$) and AAC with 66.1% ($480 \mu\text{J/cm}^2$). For the R-10 bleach, the best results were for CAAC, with 69.9% ($1280 \mu\text{J/cm}^2$), followed by the pyrogalol developer, with 68.2% ($640 \mu\text{J/cm}^2$) and AAC with 64.3% ($960 \mu\text{J/cm}^2$)[2].

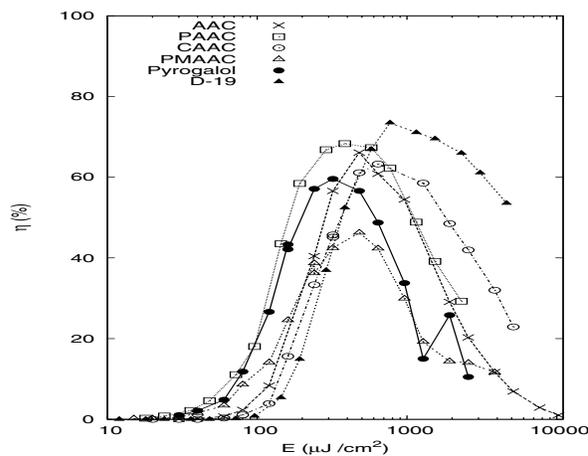


Figure 2: η -Log E curves of holographic transmission gratings recorded on emulsions BB640 developed with AAC, PAAC, CAAC, PMAAC, pyrogalol and D-19 and bleached with ferric bleach.

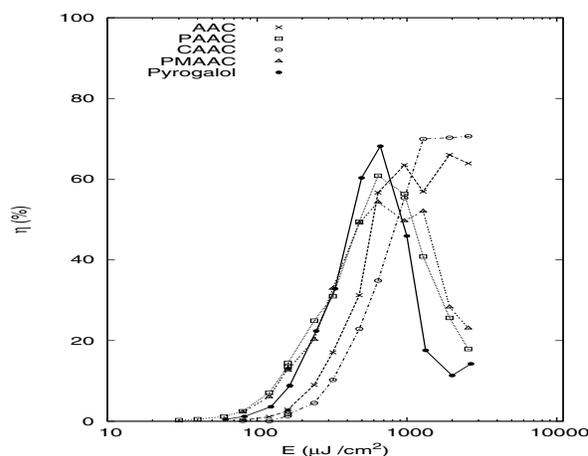


Figure 3: η -Log E curves of holographic transmission gratings recorded on emulsions BB640 developed with AAC, PAAC, CAAC, PMAAC, pyrogalol and D-19 and bleached with fixation free rehalogenating bleach R-10.

Another study was done with holographic reflection gratings using only fixation free rehalogenating bleach R-10. In this study, performed at a later stage than the transmission described above, the presensitizing was done with 3% solution of TEA, so that the exposure energy values presented will be lower than expected. Solvent and direct bleaches were rejected in this case since both processes eliminate material from the emulsion, thus causing a distortion of the grating by the shrinkage of the layer. The results are presented in fig. 4. Best values were 72,1% ($900 \mu\text{J}/\text{cm}^2$) for AAC developer, 68,3% ($600 \mu\text{J}/\text{cm}^2$) for PAAC, 71,1% ($1800 \mu\text{J}/\text{cm}^2$) for CAAC and 67,8% ($900 \mu\text{J}/\text{cm}^2$) for Pyrogalol.

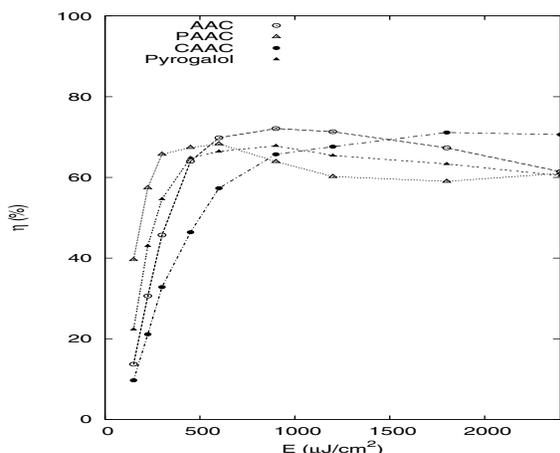


Figure 4: η -Log E curves of holographic reflection gratings recorded on emulsions BB640 developed with AAC, PAAC, CAAC and Pyrogalol, bleached with fixation free rehalogenating bleach R-10.

Special results

The above results may be considered standard for a silver halide emulsion, although the diffraction efficiency figures are quite good. Nevertheless, we have obtained results that were not found for former classical emulsions. The most remarkable of them follow.

Solvent bleach R-9

The best diffraction efficiencies for transmission gratings were not those presented in the former section, but those obtained with AAC developer and reversal bleach Kodak R-9. Values went as high as 82.9% for an exposure energy of $650 \mu\text{J}/\text{cm}^2$, and with very clear and clean recordings[2].

High densities with D-8

Another non conventional result was the very high densities obtained with the high contrast developer Kodak D8 when exposed to an holographic transmission setup. Densities obtained were above the detection limits of our measurements devices, which is 11.3. Figure 5 shows the results of the anomalous D-Log E[3] curve together with the holographic response of the direct bleached emulsion with ferricnide and of the solvent bleach R-9. Both bleaches were selected for extracting direct information from the holographic structure recorded, since they do not present diffusion processes. The analysis of the resulting DE curves, with a double peak for the direct bleach and a single maximum for the solvent bleach right in the intermediate energy zone of the direct bleach led us to postulate a new image formation mechanism[4].

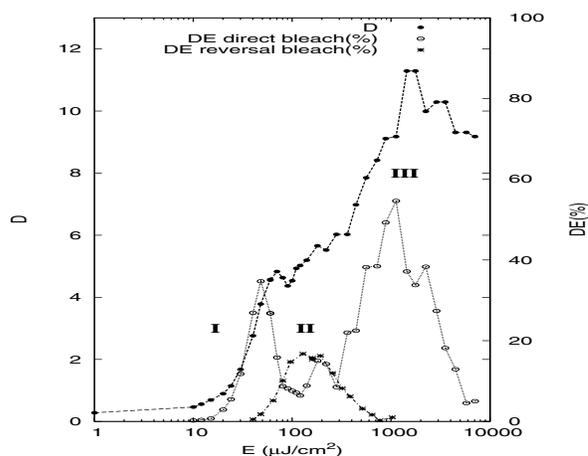


Figure 5: BB640 plates processed with Kodak D8 developer. D - Log E curve (\bullet), DE - Log E curve for non-hardening fix followed by ferricnide rehalogenating bleaching (\odot) and DE - Log E curve for Kodak R-9 reversal bleaching (\star).

Exposure of BB640 with blue

Based on the absorption band of silver halide, located near 400 nm, we have tried using red sensitive BB640 emulsion for recording reflection holograms with a He-Cd laser (wavelength 442 nm). Spatial frequency was higher than 7000 l/mm. Plates had been presensitized with 2 minutes in the 3% TEA solution followed by 6 minutes in the deionized water bath. Fig. 6 shows the spectral response of the best grating, with a DE of 50,5% at an exposure energy of $600 \mu\text{J}/\text{cm}^2$. Replay wavelength is 440.1 nm.

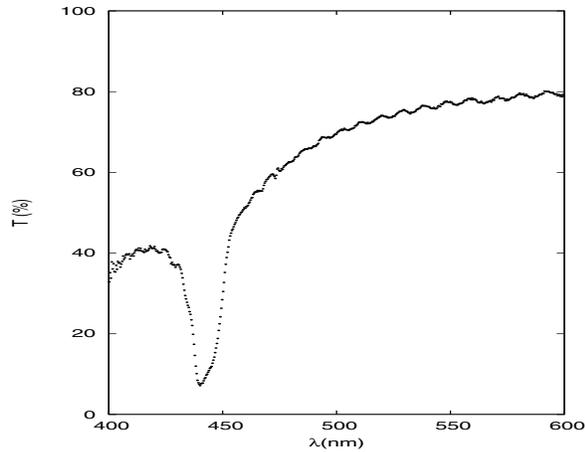


Figure 6: Spectral response of a unslanted reflection grating recorded with BB640 emulsion using a He-Cd laser (442 nm). Exposure energy is $600 \mu\text{J}/\text{cm}^2$.

Hologram multiplexing

The results obtained with the solvent bleach and the developer D8 points towards a high index modulation capability of this material. To check this properties we have tested hologram multiplexing in a single layer of emulsion. The most outstanding result is the superposition of reflection and transmission gratings in the same plate, to obtain three dimensional structures[6]. Fig. 7 shows the simulation of the refractive index of the structure obtained after multiplexing one unslanted volume reflection grating and two crossed unslanted volume transmission gratings. Diffraction efficiencies were 52% for the reflection grating and 60% for each of the transmission gratings.

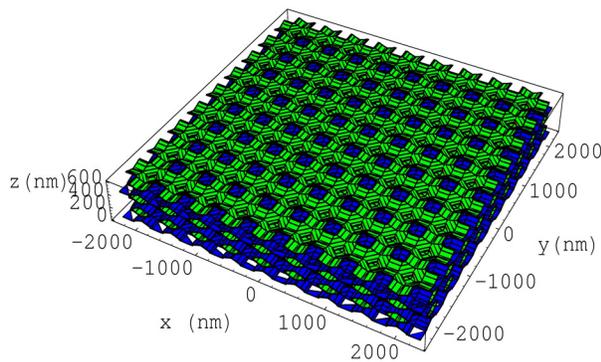


Figure 7: Simulation of the refractive index three dimensional structure obtained by multiplexing one reflection and two transmission diffraction gratings.

Panchromatic emulsions BBVPan

A still non commercial panchromatic version of BB640, denominated BBVPan, was tested for recording multiple reflection gratings by the multiplexing method with three laser wavelengths (He-Ne - 632.8 nm, Nd-Yag - 532 nm and He-Cd - 442 nm) in order to characterize it for color holography. The best result was obtained with a sequence of exposures with energies of 150 (442 nm) + 250 (532 nm) + 1200 (632.8 nm) $\mu\text{J}/\text{cm}^2$, for which the diffraction efficiencies of each band are balanced, with 52% for the red, 62% for the green and 57% for the blue wavelength, with the corresponding spectral transmission curve shown in fig. 8[5].

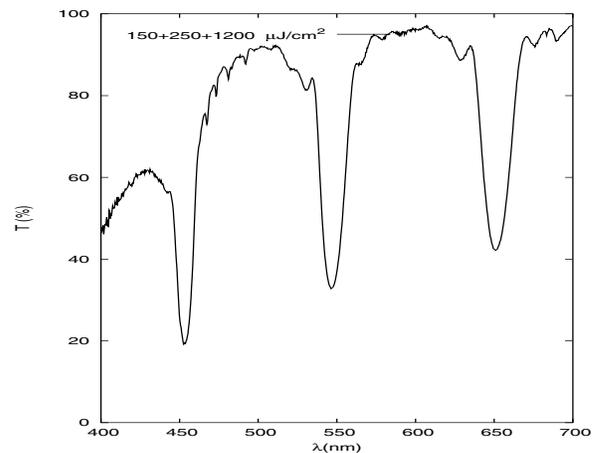


Figure 8: Transmission spectrum of the multiplexed holographic reflection grating recorded on a single BBVPan plate.

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Biography

Manuel Ulibarrena received his MS in physics from Universidad de Santiago de Compostela, Spain, in 1986 and his PhD at the Departamento de Ciencia y Tecnología de Materiales of Universidad Miguel Hernández de Elche, Spain, in 2003. He is currently an associate professor of Non-destructive Testing in the same institution. He has been involved in holographic research since 1985, with experience in holographic displays, holographic optical elements and holographic recording materials research. His current area of interest is research on silver halide holographic recording materials.