J. Modern Opt.
28 ABR. 1998
Received
5 MAYO 1998
Accepted.
8 JUN. 1998

High efficiency silver halide sensitized gelatin holograms with low absorption and scatter

A. BELÉNDEZ (†), C. NEIPP (†), M. FLORES (‡) and I. PASCUAL (‡)

(†) *Departamento de Física, Ingeniería de Sistemas y Teoría de la Señal. Universidad de Alicante. Apartado 99, E-03080 Alicante (SPAIN)*

(‡) *Departamento Interuniversitario de Óptica. Universidad de Alicante. Apartado 99, E-03080 Alicante (SPAIN)*

Phone: +34-(9)6-590 36 51
Fax: + 34-(9)6-590 34 64
E-mail: augusto@disc.ua.es

Journal of Modern Optics: ***Letter***

ABSTRACT

We report for the first time the results of a comparative performance study of silver halide sensitized gelatin (SHSG) holograms recorded in the familiar Agfa 8E75 HD plates and the new BB-640 plates manufactured by Holographic Recording Technologies. High diffraction efficiency (~90%) and low absorption and scatter (~5.4%) are the distinguishing features of the BB-640 plates, given the fact that both kinds of plates have been prepared and processed under identical conditions. Thus, in summary, our investigation reveals that high quality SHSG holograms could be obtained using the new BB-640 plates.

1. Introduction

Silver halide sensitized gelatin (SHSG) has proven to be a good alternative to dichromated gelatin (DCG) in the production of transmission holograms. SHSG is a hybrid process that combines the sensitivity of photographic emulsion with the quality of DCG. In this processing, the exposed emulsion is developed, bleached and fixed and the silver halide grains are eliminated from the emulsion and the recorded holographic image is due to the variation in the degree of hardening between the exposed and non-exposed zones of the emulsion [1]. During the bleaching process, the developer silver is oxidized to Ag^{+}, whereas the Cr^{+6} ions is reduced to Cr^{+3} during the same bleaching bath. These Cr^{+3} ions formed cross-links between the gelatin chains in the vicinity of the oxidized silver grains, causing local hardening. After the fixing step, the processing is completed with the washing of the emulsion and its dehydration by means of successive baths in isopropanol, which is similar to the processing of DCG. This technique in its present-day state is the result of the work done by Graver *et al.* [2] on the one hand, and Chang and Winick [3] on the other. Kodak 649F emulsions were used in the first studies on SHSG holograms [2-5]. During recent years some optimized procedures for SHSG holograms derived from Agfa plates [6-8] have been published, showing that this emulsion is suitable for obtaining SHSG transmission holograms.

One of the photographic emulsions mainly used in Holography were made by Agfa Gevaert. These emulsions have been very highly regarded during the last decades. The fact that Agfa holographic materials will no longer be produced is of great concern to many in Holography. This implies that it is necessary to find and to study alternative silver-halide materials, in particular, for SHSG holograms. One of the available silver-halide materials is the new and unfamiliar BB-640 emulsion from Holographic Recording Technologies*. However, to the best of our knowledge no papers on this new material have been published, except for the information provided by the manufacturer [9].

In this paper we present the results of a comparative investigation between the performance of SHSG formed out of BB-640 and Agfa 8E75 HD plates. The influence of the hyper-sensitization with solution containing sodium sulfite and urea, the effect of bleach temperature and the drying in low humidity enviroment have been investigated and their influence on recording sensitivity, diffraction efficieny ~~(diffraction efficiency)~~ and absorption and scatter are analyzed and discussed.

2. Experimental

Experiments were carried out with BB-640 plates and with Agfa 8E75 HD plates, in order to compare the results obtained. Unslanted holographic transmission-diffraction gratings were recorded on each plate by using two collimated beams from a 15 mW He-Ne laser (632.8 nm) making an angle of 45° (in air) with each other. With the geometry described, the spatial frequency of the gratings was calculated as 1209 lines/mm. The total intensity was ~500 μW/cm^2 and the beam intensity ratio was 1:1.

BB-640 emulsions have a low grain size between 20 nm and 25 nm [9] (common Agfa-Gevaert 8E75 HD material has a mean grain size in the uncoated emulsion of 35 nm and the finished coated product has grains of about 40 nm to 45 nm [10]). However, the sensitivity of BB-640 plates is lower than that of Agfa 8E75 HD plates. Firstly, amplitude holograms were recorded and D-log E curves were obtained for BB-640 and Agfa 8E 75 HD plates exposed at 632.8 nm, developed with D-19 developer and fixed in F-24, a non-hardening fixer. It was found that BB-640 developed to a density of 0.6 [1] have a sensitivity of 214 μJ/cm^2, while for Agfa 8E75 HD plates the sensitivity is 5.5 μJ/cm^2. Also, BB-640 emulsions are hardened to a high degree [9] and their gelatin is too hard to be used directly for recording SHSG holograms. We solved the problem of the high degree of initial hardening of the gelatin emulsion by soaking the unexposed plates in a weak fixer. A series of plates were pretreated by immerzing in a solution of distilled water with a sodium sulfite concentration of 1% and an urea concentration of 5% (by weight) for 10 minutes at 20° C. The plates were then rinsed in running water for 1 minute and dried for 24 hours at 20° C and 60% RH. This pretreatment produces not only a softening of the gelatin of the BB-640 emulsion but also an increase in the sensitivity by a factor of ~2.6 and a significant increase in diffraction efficiency.

Next, phase holograms were recorded using the same recording geometry. The exposed plates were processed according to the processing schedule shown in Table I. This processing is a modification of the processing presented in reference 6: hypersensitizing the plates before exposure, increasing the time of washing of the plates after development and bleaching (in order to be sure to eliminate residual chemicals in the layer), increasing the temperature of the bleach bath from 50° C to 70°C, and drying the processed plates in a dissecator at low relativity humidity during 24 hours instead of using a vacuum chamber. The developer used in these experiments was Kodak D-19, a nontannning developer, although it is important to note that oxidation products of this developer produce an important tanning action [4].

3. Results and discussion

The diffraction efficiency η of the recorded holograms was calculated as the ratio of the diffracted beam intensity to the incident collimated probe-beam intensity of the He-Ne laser, taking into account Fresnel losses and absorption due to the glass substrate. The efficiency of zero-order τ was calculated similarly as the ratio of the directly transmitted beam intensity to the incident power, corrected taking into account losses due to reflection at different interfaces and absorption of the glass substrate. The diffraction efficiency as a function of exposure energy is shown in Fig. 1 where the results for BB-640 with and without pretreatment and for two bleach temperatures are presented. As we can see in this figure a very high diffraction efficiency of 90.3% (79% if we do not consider Fresnel losses and the absorption of the glass plate) was obtained and the diffraction efficiency is higher using hyper sensitized plates than in the case of normal plates. While for hyper sensitized plates it is necessary to increase the temperature of the bleach bath to obtain the best results, normal plates bleached at 50° C show better results than the same plates bleached at 70° C. Fig. 2 shows the measured diffracted and transmitted beam intensities for replay on Bragg and for pretreated BB-640 and Agfa 8E75 HD plates bleached at 70° C. As can be seen, BB-640 shows better results with a diffraction efficiency of 90.3% and a zero-order efficiency of 4.3%. This implies that of the light incident on the emulsion, only 5.4% remains unaccounted for, lost through scatter and absorption. For Agfa plates diffraction efficiency is 84%, transmission is 0.7% and light unaccounted for is 15.3%, almost three times more than for BB-640 plates.

For BB-640 plates the maximum achieved diffraction efficiency of 90.3% is limited almost solely by absorption and scatter. In order to obtain a value for absorption and scatter losses, a percentage as a function of exposure was calculated as $100 - \eta - \tau$, where η is the diffraction efficiency and τ is the efficiency of zero-order, both measured at Bragg angle. Fig. 3 shows the results obtained for BB-640 and Agfa 8E75 HD plates. Absorption resulting from the stain and scatter measured in processed SHSG from Agfa 8E75 HD holographic plates without treatment before exposure takes values from 7% to 17% for a bleach bath temperature of 50° C and from 15% to 22% for 70° C. For Agfa plates with pretreatment, the values are between 8% and 16% for bleach bath at 50° C and between 15% and 31% for 70° C. These results show that absorption and scatter increase with the bleach temperature and are a function of the exposure. However, for BB-640 plates the total losses caused by absorption and scatter were as low as 3-7% for all cases. No increase in absorption and scatter was observed within the exposed interval. It is also clear that the lower absorption and scatter of BB-640 plates allows higher diffraction efficiencies to be obtained. For SHSG in BB-640 emulsion and for an absorption and scatter equal to 5.4%, the maximum diffraction efficiency possible is ~95%, and we have obtained ~90%. We think that the good results obtained for BB-640 plates are due to their lower grain size, although both SHSG holograms from BB-640 and Agfa 8E75 HD emulsions are clear and transparent with little stain, scattering is much higher for Agfa as can be seen on a visual inspection of the processed plates by reconstructing them using a beam directly from the laser.

Measurements were also made of the scattered light intensity using the set-up shown in Fig. 4., while Fig. 5 shows the results obtained for BB-640 and Agfa 8E75 HD plates. In this figure scattered light intensity due to the glass substrate of Agfa 8E75 HD and BB-640 plates has been included. Values of ~1 and ~1.2, in arbitrary units, have been obtained for BB-640 and Agfa glass substrata, respectively. Scattered light intensity measured in processed SHSG from Agfa 8E75 HD holographic plates without treatment before exposure takes values from 3 to 46 for a bleach bath temperature of 50° C and from 9 to 62 for 70° C. For Agfa plates with pretreatment, the values are between 9 to 68 for bleach bath at 50° C and between 19 to 87 for 70° C. However, for BB-640 plates the total losses caused by scatter were as low as ~2-10 for all cases. As can seen in this figure scattered light intensity for BB-640 plates is lower than for Agfa plates and the maximum value for scattering in SHSG holographic gratings of the Agfa emulsion is almost nine times the maximum scattering obtained using the BB-640 emulsion.

In Table II we have summarized the experimental results obtained for replay on Bragg for pretreatred BB-640 and Agfa 8E75 HD plates bleached at 70° C. In this table the values correspond to the value of exposure for maximum diffraction efficiency.

The results obtained for diffraction efficiency for SHSG holograms derived from BB-640 holographic plates and presented in this paper are the best reported at the present time. For hyper sensitized Agfa 8E75 HD emulsion bleached at 70° C following the same procedure as for BB-640 plates, we obtained a maximum diffraction efficiency of 84% but the absorption and scatter was 15.3%. In a previous paper [8], for SHSG transmission holograms derived from Agfa emulsions with a spatial frequency of ~1200 lines/mm and for D-19 developer, a diffraction efficiency of ~84% was obtained taking into account losses caused by reflection. Simova and Kavehrad [7] obtained a diffraction efficiency of ~80% for the same emulsion, spatial frequency and developer, bleached at 50° C. They measured total losses caused by absorption and scatter of ~10-15%, similar to the values that we have obtained for the same emulsion and bleach temperature.

BELÉNDEZ, Augusto, et al. "High-efficiency silver-halide sensitized gelatin holograms with low absorption and scatter". Journal of Modern Optics. Vol. 45, No. 10 (Oct. 1998). ISSN 0950-0340, pp. 1985-1992 DOI: 10.1080/09500349808231736

4. Conclusions

In summary, our results have demonstrated the possibility of recording high quality transmission holograms in SHSG using the novel BB-640 emulsion. After some trials we found that the diffraction efficiency increases significantly by using hyper sensitized plates by means of a weak fixing solution, which produces better results than normal plates. Pretreatment significantly increased not only the diffraction efficiency (which reached ~90%) but also the energetic sensitivity (which increased ~2.6 times). Another significant improvement was discovered when the bleach-bath temperature was increased from 50° C to 70° C. This increase in temperature improves the diffraction efficiency but does not produce an increase in absorption and scatter, as opposed to what happens in the case of the Agfa 8E75 HD emulsion. SHSG holograms formed on BB-640 emulsion show a high diffraction efficiency and a low value for light lost through absorption and scatter. These losses are lower than for SHSG holograms formed on Agfa 8E75 HD emulsion. The very high diffraction efficiency and low absorption and scatter obtained certainly breaks new ground with regard to results obtained in experiments with SHSG holograms over the past years. The results presented in this paper are one of the best reported for this type of processing technique at the present time and they confirm the future application of SHSG derived from BB-640 plates for recording high quality phase transmission holograms. The results also show that BB-640 plates are a potencial replacement for Agfa 8E75 HD emulsion in a crucial moment in which the fact that Agfa will cease its production of holographic material soon and this implies that it is necessary to find alternative silver-halide materials. Finally, the information presented in this paper in regard to the performance behavior of BB-640 plates might be useful to the researchers working in the areas of display holograms, holographic optical elements and holographic data storage.

**HRT Holographic Recording Technologies GmbH. Am Steinaubach 19, 36296 Steinau, Germany.*

Acknowledgments

M. Flores wishes to acknowledge a scholarship granted by the "Agencia Española de Cooperación Internacional (AECI)".

BELÉNDEZ, Augusto, et al. "High-efficiency silver-halide sensitized gelatin holograms with low absorption and scatter". Journal of Modern Optics. Vol. 45, No. 10 (Oct. 1998). ISSN 0950-0340, pp. 1985-1992 DOI: 10.1080/09500349808231736

REFERENCES

[1] BJELKHAGEN, H. I., 1993, *Silver Halide Recording Materials for Holography and Their Processing* (Springer-Verlag, Berlin).

[2] GRAVER, W. R., GLADDEN, J. W. and EASTES, J. W.,1980, *Appl. Optics* **19**, 1529.

[3] CHANG, B. J. and WINICK, K., 1980, *Proc. SPIE* **215**, 172.

[4] HARIHARAN, P., 1986, *Appl. Optics* **25**, 2040.

[5] ANGELL, D. K., 1987, *Appl. Optics* **26**, 4692.

[6] FIMIA, A., BELÉNDEZ, A. and PASCUAL, I., 1991, *J. Mod. Optics* **38**, 2043.

[7] SIMOVA, E. S. and KAVEHARAD, M., 1994, *Appl. Optics* **33**, 1875.

[8] FIMIA, A., PASCUAL, I. and BELÉNDEZ, A., 1995, *Opt. Laser Tech.* **27**, 285.

[9] BIRENHEIDE, R., 1997, *Holography* **8** (1), 12.

[10] PHILLIPS, N. J., HEYWORTH, H. and HARE, T, 1984, *J. Photogr. Science* **32**, 158.

BELÉNDEZ, Augusto, et al. "High-efficiency silver-halide sensitized gelatin holograms with low absorption and scatter". Journal of Modern Optics. Vol. 45, No. 10 (Oct. 1998). ISSN 0950-0340, pp. 1985-1992 DOI: 10.1080/09500349808231736

FIGURE CAPTIONS

Fig. 1.- Diffraction efficiency of SHSG transmission gratings versus exposure for BB-640 holographic plates.

Fig. 2.- Measured diffracted and transmitted beam intensities versus exposure for replay on Bragg and for pretreated BB-640 and Agfa 8E75 HD plates bleached at 70° C.

Fig. 3.- Absorption and scatter of SHSG transmission holographic gratings versus exposure for BB-640 and Agfa 8E75 HD plates.

Fig. 4.- Experimental set-up for the measurement of scattered radiation.

Fig. 5.- Variation of the scattered light intensity with exposure for SHSG gratings recorded using BB-640 and Agfa 8E75 HD plates.

TABLES

Table I.- Processing schedule.

Table II.- Diffraction efficiency, absorption and scatter, and scattered light intensity for replay on Bragg for the value of exposure corresponding to maximum efficiency.

BELÉNDEZ, Augusto, et al. "High-efficiency silver-halide sensitized gelatin holograms with low absorption and scatter". Journal of Modern Optics. Vol. 45, No. 10 (Oct. 1998). ISSN 0950-0340, pp. 1985-1992 DOI: 10.1080/09500349808231736

TABLE I

A. Hyper sensitization

1. Soak plates in 1% sodium sulfite and 5% urea solution for 10 min at 20°C.

2. Rinse in running water for 1 min.

3. Dry for 24 h at 20° C and RH = 60%.

B. Exposure

C. Development

1. Develop in D-19 for 5 min.

2. Rinse in running water for 2 min.

3. Bleach for 30 s after the plate has cleared at 70° C.

4. Rinse in running water for 2 min.

5. Soak in nontanning fixer F-24 for 2 min.

6. Wash in running water for 10 min.

8. Dehydrate in 50% isopropanol for 3 min.

9. Dehydrate in 90% isopropanol for 3 min.

10. Dehydrate in 100% isopropanol for 3 min.

11. Dry in dissecator for 24 h at 20° C and RH < 20%.

All solutions are at 20° C except the bleaching step.

Bleach formula

Solution A:	Ammonium dichromate	20 g
	Sulfuric acid	14 mL
	Distilled water	1 L
Solution B:	Potassium bromide	92 g
	Distilled water	1 L

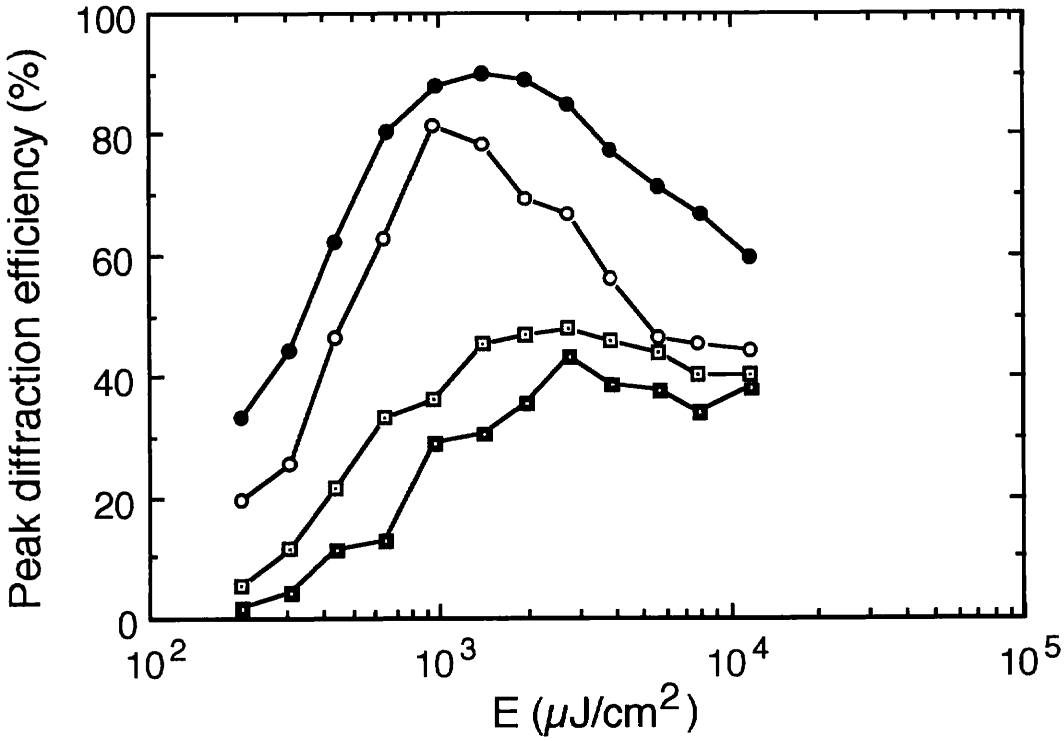
Just before use, mix one part A with ten parts distilled water, then add 30 parts B.

BELÉNDEZ, Augusto, et al. "High-efficiency silver-halide sensitized gelatin holograms with low absorption and scatter". Journal of Modern Optics. Vol. 45, No. 10 (Oct. 1998). ISSN 0950-0340, pp. 1985-1992 DOI: 10.1080/09500349808231736

TABLE II

	BB-640	Agfa 8E75 HD
Peak diffraction efficiency (%)	90.3	84.0
Absorption and scatter (%)	5.4	15.3
Scattered light intensity (arbitrary units)	3.5	~~17.8~~ 30.

BELÉNDEZ, Augusto, et al. "High-efficiency silver-halide sensitized gelatin holograms with low absorption and scatter". Journal of Modern Optics. Vol. 45, No. 10 (Oct. 1998). ISSN 0950-0340, pp. 1985-1992 DOI: 10.1080/09500349808231736



Normal plates (bleach: —□— T = 50° C, —■— T = 70° C)

Pretreated plates (bleach: —○— T = 50° C, —●— T = 70° C)

FIG. 1

BELÉNDEZ, Augusto, et al. "High-efficiency silver-halide sensitized gelatin holograms with low absorption and scatter". Journal of Modern Optics. Vol. 45, No. 10 (Oct. 1998). ISSN 0950-0340, pp. 1985-1992 DOI: 10.1080/09500349808231736

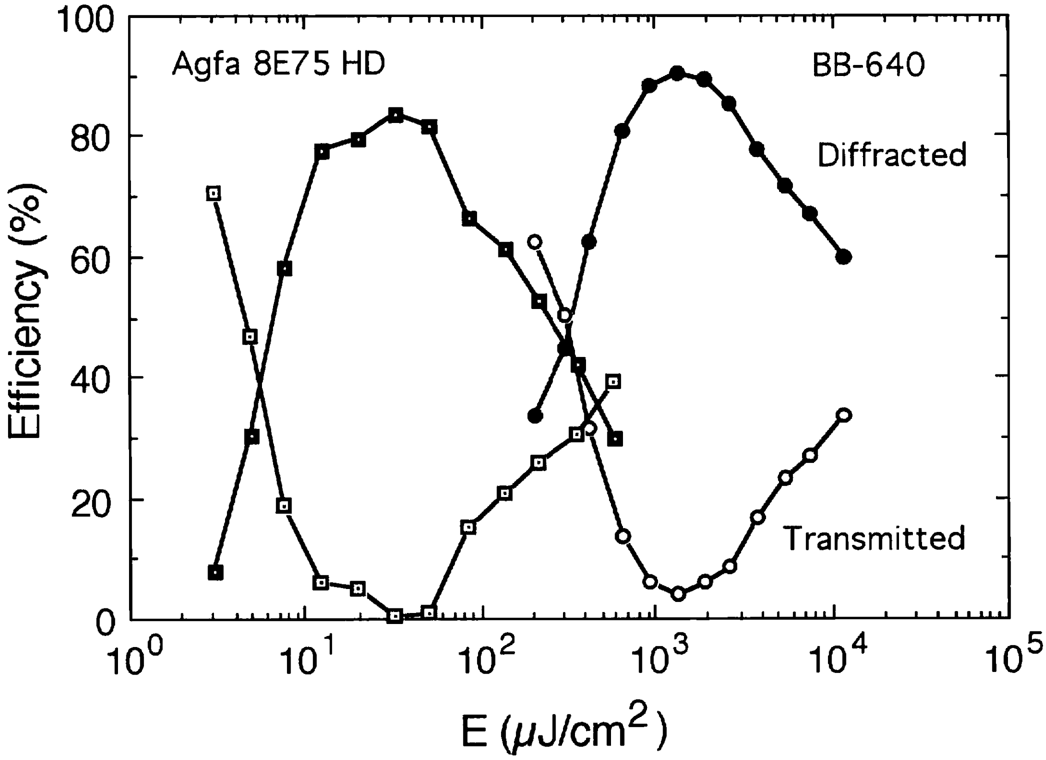
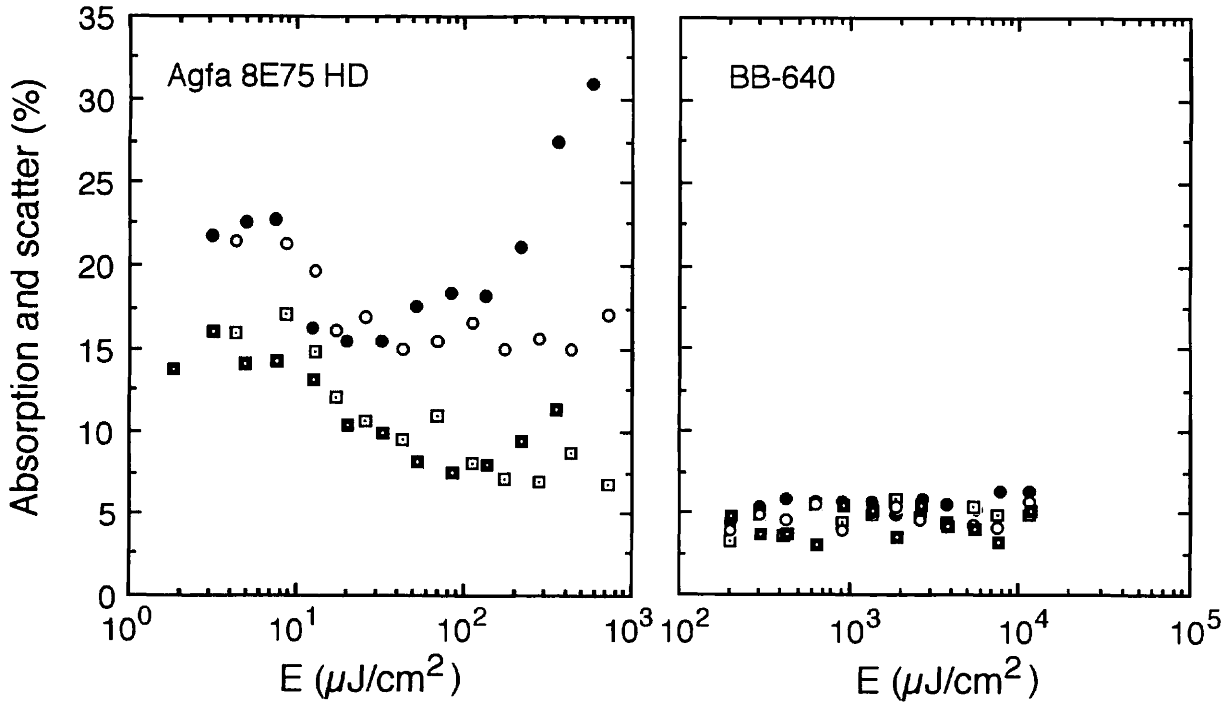


FIG. 2

BELÉNDEZ, Augusto, et al. "High-efficiency silver-halide sensitized gelatin holograms with low absorption and scatter". Journal of Modern Optics. Vol. 45, No. 10 (Oct. 1998). ISSN 0950-0340, pp. 1985-1992 DOI: 10.1080/09500349808231736



Bleach temperature, T = 50°C (pretreated plates, ■ ; normal plates, □)
Bleach temperature, T = 70°C (pretreated plates, ● ; normal plates, ○)

FIG. 3

BELÉNDEZ, Augusto, et al. "High-efficiency silver-halide sensitized gelatin holograms with low absorption and scatter". Journal of Modern Optics. Vol. 45, No. 10 (Oct. 1998). ISSN 0950-0340, pp. 1985-1992 DOI: 10.1080/09500349808231736

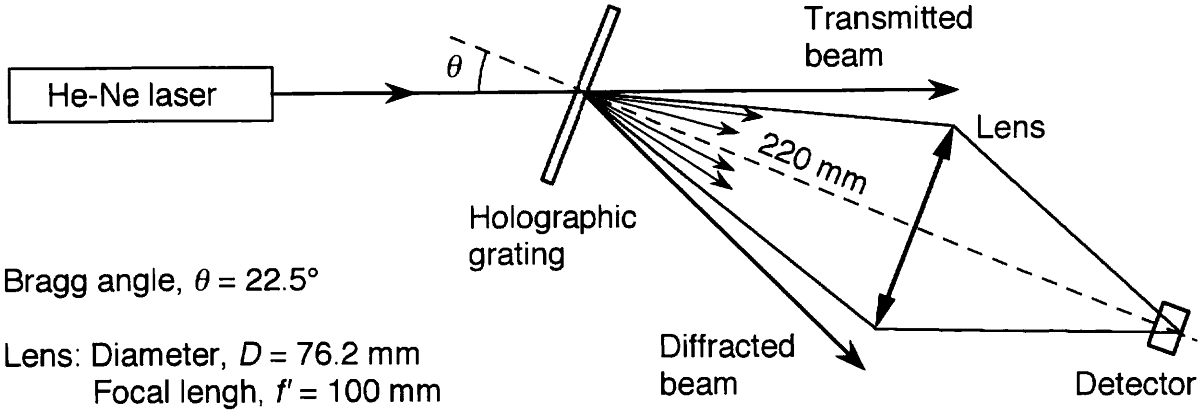
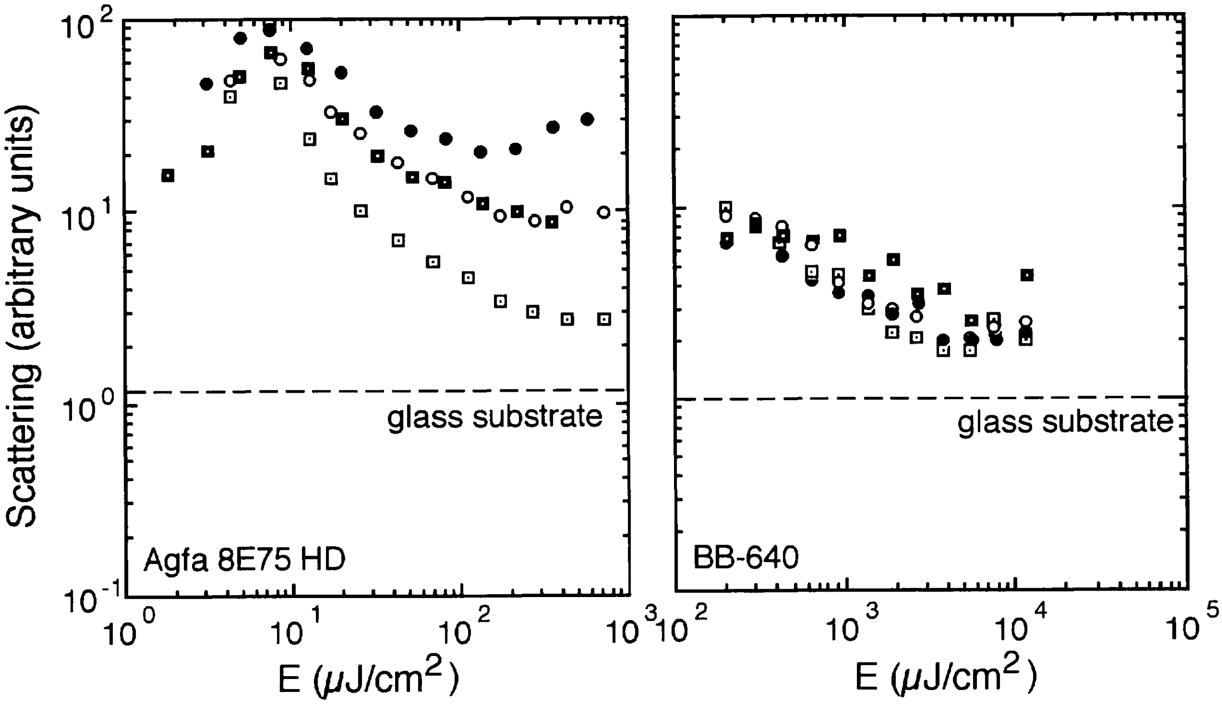


FIG. 4

BELÉNDEZ, Augusto, et al. "High-efficiency silver-halide sensitized gelatin holograms with low absorption and scatter". Journal of Modern Optics. Vol. 45, No. 10 (Oct. 1998). ISSN 0950-0340, pp. 1985-1992 DOI: 10.1080/09500349808231736



Bleach temperature, T = 50°C (pretreated plates, ■; normal plates, □)

Bleach temperature, T = 70°C (pretreated plates, ●; normal plates, ○)

FIG. 5