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# Changes of the diffraction efficiency due to emulsions thicknesses in holographic gratings

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#### Abstract

We analyze the behavior of the diffraction efficiency as a function of the thickness of the relief holographic grating recorded on a phase emulsion composed by rosin and bromophenol blue (BPB) dye. The emulsions thicknesses are mainly due to the rosin quantity deposited on a substrate. We record holographic gratings on each emulsion using the spectral line  $\lambda = 457$  nm of an argon laser, after this we developed the emulsion with a quick process. The diffraction efficiencies for each grating vary from 0.25% to 0.62%. © 2006 Elsevier B.V. All rights reserved.

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# 1. Introduction

In order to characterize holographic materials, it is commonly used to make phase or amplitude diffraction gratings due to its easiness to record and measure the characteristic parameter of the material, the diffraction efficiency  $\eta(\%)$ ; because it can be related with the relief grating modulation and some other parameters such as the exposure energy and/or the developing time.

The desired relief of a phase holographic grating can be obtained by a convenient choice of the thickness of the emulsion or holographic film. It has been reported that different concentrations between the elements that compose the material or film, produce different thicknesses allowing a change in the relief deep,  $\Delta d$ , of the diffraction gratings

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and it modifies the phase change,  $\phi(x, y)$ , of a light beam passing through the grating [1].

For phase holograms, the phase changes are proportional to  $\Delta d$  and it is a function of the exposure energy density and the developing time [2], as is shown in

$$\Delta d = T \left[ r_1 - \Delta r \mathrm{e}^{-cE} \right],\tag{1}$$

where T is the developing time,  $r_1$  the removing speed of material,  $\Delta r$  the speed difference between  $r_1$  and the non-affected material, c is a constant and E the exposure energy.

This relief depth is related, as is indicated in Eq. (2), to the phase change  $\phi(x, y)$  and the phase holograms transmittance function T(x, y),

$$T(x, y) = e^{i\phi(x, y)} = e^{ikn\Delta d},$$
(2)

where *n* is the film refraction index, and *k* which relates with the wavelength  $(k = 2\pi/\lambda)$  is a constant called wave number.

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We propose the characterization of a holographic material as a function of its thickness and as a function of their concentrations. The material we use is composed by rosin and bromophenol blue dye (BPB) mixed with isopropyl alcohol which allow us to record the phase holographic grating.

The rosin is obtained from Oocarpa Mexican pines, is insoluble in water, freely soluble in alcoholic solutions as benzene, ether, abietic acid, oils, carbon disulfide and can be fixed in alkaline hydroxides; it has many applications [3,4] and it was employed as a holographic material mixed with other dyes [5–7].

The BPB dye is commonly used as a pH chemical indicator sensing, and has biomedical, biological and chemical engineering applications. BPB dye is soluble in ethylic alcohol and benzene freely soluble in water [8–11].

The different concentrations are obtained when we change the quantities between the rosin and BPB dye, so we obtain different densities for each emulsion. We prepare several emulsions with different concentrations to obtain several thicknesses and characterize the material measuring the  $\eta(\%)$  of the gratings recorded on it. We found the optimal thickness of the emulsion for obtaining the best  $\eta(\%)$  for the gratings.

There are some other papers about thickness of the films. Dzyubenko et al. [12] studied the behavior of the  $\eta(\%)$  as a function of the shrinkage of the silver-halide holographic material Agfa Gevaert 8E75. In [13,14] reported the reconstruction of the images of the holograms recorded in silver halide films 649F and 10E70, having a distortion of the images due to thickness variations of the film and was found to have an average of 20%.

### 2. Preparation of the emulsions

The process for the preparation of the holographic material was made by mixing different concentrations of rosin and BPB dye diluted with isopropyl alcohol. We deposited by the gravity technique the same quantity of each emulsion on a  $2 \times 2$  in glass plate previously cleaned; we let the emulsion in repose during 12 h and then introduced in an oven at 100 °C during 3 h, after which, the emulsion became hardened and the isopropyl alcohol was evaporated.

In Table 1 we show the concentrations for the emulsions keeping the same isopropyl alcohol and BPB proportion on each case, and we only change the rosin quantity, this let us obtain different thicknesses, where the major number indicates the rosin and the smaller number indicates the dye.

Table	1							
Rosin	and	BPB	dye	conc	entrat	ions	relati	on

Concentration	10:1	20:1	30:1	40:1	80:1	
Rosin (g) BPB (g)	0.30 0.03	0.60 0.03	0.90 0.03	1.20 0.03	2.40 0.03	
Alcohol (ml)	5	5	5	5	5	



Fig. 1. Absorbance spectrum curves vs. wavelength for different concentration of rosin and BPB. In the UV region this material correspond to higher absorbance.

Fig. 1 shows five curves of absorbance of the emulsions shown in Table 1. Observe that in the line at  $\lambda = 457$  nm, which was the wavelength used to record all the gratings, the absorbance increased from 0.25% to 1%. To obtain these curves, a Perkin–Elmer  $\lambda 3$  spectrophotometer was used.

## 3. Results

Once the different emulsions are obtained we measure the film thickness by a Federal<sup>®</sup> Surfanalizer 400 profilometer. The measurements of the five samples mentioned in Table 1 are shown in Fig. 2, where the vertical axis represents its thickness in microns ( $\mu$ m) with an error of  $\pm 0.5 \mu$ m; and the horizontal axis is the distance carried



Fig. 2. Different emulsions thickness (l) in  $\mu$ m with the surfanalizer 400 measurements.



Fig. 3. Emulsion concentrations between its thicknesses relations with polynomial fitting.

for the profilometer on the sample. We can see in Fig. 2 that the 80:1 concentration has a bigger thickness; this emulsion contains more rosin than other emulsions. The thicknesses are between 4 and  $27 \,\mu\text{m}$  approximately.

In Fig. 3 we show the relation between the emulsion concentration and its thickness obtained experimentally (see Fig. 2) as a linear relation indicated by square symbols of real data and a polynomial analysis of the data which gives us Eq. (3) indicated by circle symbols that predict the thickness (*Y* vertical axis), as a function of the emulsion distance (*X* horizontal axis), where we do not have the lineal and polynomial fitting of some differences in this case.

$$Y = 0.90512 + 0.33426X - 1.04551E - 4X^2.$$
(3)

We prepare more emulsions with different concentrations that have been mentioned previously. Considering Eq. (3) we can obtain more thicknesses for emulsions that were not physically measured. In Table 2 we show the concentrations and its thickness obtained.

In Table 2, the bold letter represents the concentration measure and the normal letters are the concentrations and its thickness obtained with Eq. (3).

The diffraction gratings were recorded in the emulsions following the experimental setup shown in Fig. 4, where the interference angle  $\theta$  was fixed in 10°, the intensities ratio between the two beams was equal to one, and we employed the emission line of  $\lambda = 457$  nm corresponding to an argon laser.

After the recording process, we put the emulsion into the developer process for a period of 18 s. The developer is composed of water and chlorine in proportion 50:1 respectively (the emulsion do not require fixed process), then we



Fig. 4. Experimental setup employed to record holographic gratings; BS: beam splitter,  $M_1$  and  $M_2$ : mirrors.



Fig. 5. The diffraction efficiency vs. exposure energy, note that for 8  $\mu$ m is more photosensitive to low energy.

dry the emulsion. It should be noted that the developing process is fast, and does not require a dark room.

The next step, we measure the diffraction efficiency for each grating. In Fig. 5 we show the obtained results for the thickness indicated in Table 2.

We can see that the thicknesses between 8 and 14 µm corresponding to 20:1, 30:1 and 40:1 concentration (see Fig. 2), are the best suitable for making diffraction gratings, because their  $\eta(\%)$  are higher than the other thicknesses. As can be seen from Fig. 5,  $\eta(\%)$  is from 0.23% to 0.62% range and for the 4.22 and 26.35 µm thicknesses it is smaller than 0.3%.

When we achieve the developed process after the grating was recorded in the emulsion, we obtain a sinusoidal phase relief grating. To proof this, we aluminize the emulsion with this grating (emulsion center) and we impinge a laser beam and observe the diffracted orders as is shown in Fig. 6. There is no light through the emulsion due to the grating which is reflective for the aluminum coating.

It is important to indicate that we obtain a surface relief modulation in the emulsion, as it has to be dependent

Relation of between concentrations and thicknesses

Table 2

Concentration	10:1	17:1	20:1	25:1	30:1	40:1	50:1	60:1	70:1	80:1	
Thickness µm	4.22	7.5	8	9	10.68	14	17.5	20.7	23.6	26.35	



Fig. 6. Effect of reflection of aluminum layer in the sinusoidal phase relief grating showing the diffraction pattern of the surface of emulsion.

directly on the thickness, as it was indicated in other similar works in thin photopolymer layers [15,16].

Another important parameter we make to characterize the emulsion was the developing time as is indicated in Eq. (1), this modifies the grating relief. From Fig. 5 we can see that the emulsion has a higher diffraction efficiency in the concentration 30:1 that was used to record the grating for this process. We must mention that all gratings in this process were recorded with an interference angle of  $20^{\circ}$ , and with the same exposure energy and beam intensities. In Fig. 7 we have shown the diffraction efficiencies as a function of the developing time.

As can be observed from Fig. 7, the major  $\eta = 0.47\%$  is obtained at 24 s and produces the best grating relief. Note that for different developing time there are different relief modulations.

Finally, in Fig. 8 we show the photography of the diffraction grating which has been 600 times amplified by an optical microscope, the diagonal line is due to crash in



Fig. 7. This graph shows the diffraction efficiency vs. the developing time. At 24 s, we obtain  $\eta = 0.47\%$ .



Fig. 8. Diffraction grating recorded in the emulsion of rosin and BPB dye.



Fig. 9. The (-1, 0, and 1) diffracted orders from the diffraction grating.

our emulsion and, in Fig. 9 we show the diffraction pattern of this grating; in the centre is the zero diffraction order, while on either side of it, the +1 and -1 diffraction orders are shown.

## 4. Conclusions

The results shown above indicate us the best choice for our emulsion to obtain the major diffraction efficiency depending on the thickness of the emulsion and developing time. We observed that the emulsion thicknesses are directly proportional to the quantity of rosin and can be considered as important characteristics for the photosensitive holographic materials. The best diffraction efficiency was obtained with the concentration 30:1, which give us a thickness of 10.48 µm and a  $\eta = 0.47\%$ .

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