Direct measurement of the refractive index profile of phase gratings, recorded in silver halide holographic materials by phase-contrast microscopy

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Plane-wave phase holograms recorded in Agfa-Gevaert 8E75HD emulsions and processed by the combination of AAC developer and the R-9 bleaching agent were studied by phase-contrast microscopy, using high-power immersion (100×) objective. Thus the modulation of the refractive index as a function of the bias exposure and the visibility of the recording interference pattern can also be determined. Measured diffraction efficiencies were compared to those predicted by coupled wave theory, using the measured refractive index modulations. Direct measurement of the phase profile of the gratings can be used for optimizing processing. © *2003 American Institute of Physics.* [DOI: 10.1063/1.1629794]

Phase contrast microscopy was invented by Zernicke.[1] The method was applied to various fields of optical metrology and especially to biology, and it was considerably improved by Zernicke himself and others.[2–7] Contributions and improvements to the method by Françon[4,6,7] were especially important. For the study of holographically recorded phase gratings interference microscopy was also used.[8–12]

There can be found only a couple of articles on the use of phase-contrast microscopy for the study of phase gratings. One of them is the work of Kostuk and Goodman.[13] They proved the existence of a spatial frequency dependent diffusion process in the fixation-free rehalogenating bleaching of holograms recorded in Agfa 8E75HD emulsion at low (up to 100 lp/mm) spatial frequencies.

Systematic study of phase gratings, fabricated via ion implantation in glass, was performed by the author and his co-workers, both with interference- and phase-contrast microscopy.[14,15] Semiphysically developed phase holograms in Agfa 8E75HD emulsion were also studied by the author using phase-contrast microscopy.[16]

The aim of the present work was to measure the $\Delta n(E_0,V)$ characteristics of a holographic material, i.e., the dependence of the refractive index modulation of the holographic gratings on bias exposure and fringe visibility for fixed spatial frequency and processing, using phase-contrast microscopy.

Detailed description of the recording conditions and the diffraction efficiency measurements were published earlier.[17,18] They are resumed briefly here. Plane wave holograms were recorded in Agfa-Gevaert 8E75HD plates with a helium–neon laser operating at 632.8 nm. The spatial frequency of the gratings was $\nu=1200$ lp/mm. Holograms at seven values of fringe visibility, namely at $V=0.2$, 0.4, 0.6, 0.8, 0.9, 0.95, and 1.0 were recorded. Twelve holograms at exposures ranging from 10 μJ/cm^2 to 1.3 mJ/cm^2 were recorded at each visibility. Holograms have been developed with AAC developer and bleached in and R-9 solvent bleach. Diffraction efficiency of each hologram was measured. The following analytical function[19] was fitted to the measured Lin curves:[20]

$$\sigma(E_0,V)=f(E_0)(1-e^{-V})e^{\frac{-[V-V_0(E_0)]^2}{w^2(E_0)}}, \tag{1}$$

where σ is the square root of the diffraction efficiency, E_0 is the bias exposure, V is the visibility of the interference fringes and $f(E_0)$, $V_0(E_0)$, and $w(E_0)$ are parameter functions of the following form:

$$Par(E_0)=ci_{01}\left(\frac{1}{e^{\frac{ci_{1\Gamma}E_0}{ci_{12}}}+1}+ci_{13}\right)\left(\frac{1}{e^{\frac{E_0-ci_{21}}{ci_{22}}}+1}+ci_{23}\right)\times\left(\frac{1}{e^{\frac{ci_{3\Gamma}E_0}{ci_{32}}}+1}+ci_{33}\right), \tag{2}$$

where Par stands for f, $V0$ and w and ci_{xx} $(x=1,2,3)$ represent three sets of constants $(i=f,V,W)$.

Measured and fitted values of the Lin curves $[\sigma(E_0,V)]$ at five values of V are shown in Fig. 1. Values of the param-

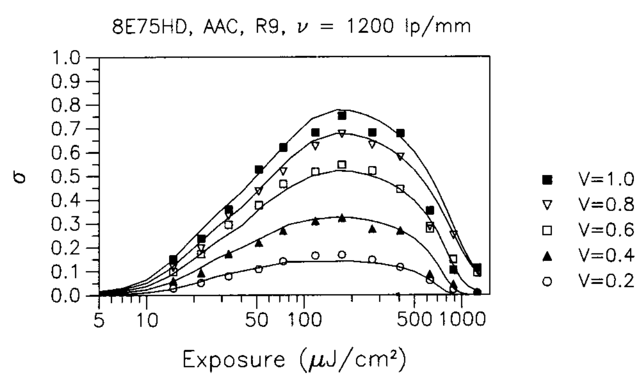

FIG. 1. Measured and fitted $\sigma(E_0,V)$ curves of Agfa 8E75HD, $\upsilon=1200$ lp/mm.

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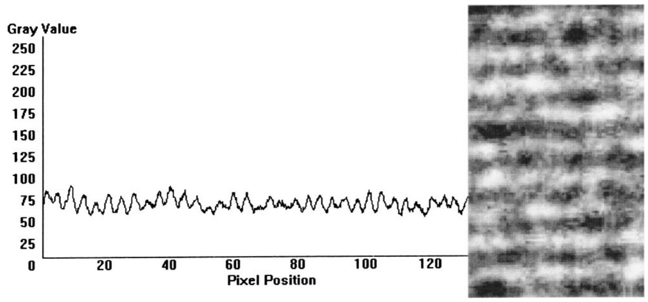

FIG. 2. Phase contrast micrograph of a hologram, point No. 6 of the $V=0.2$ curve on Fig. 1. Microscope objective=100×.

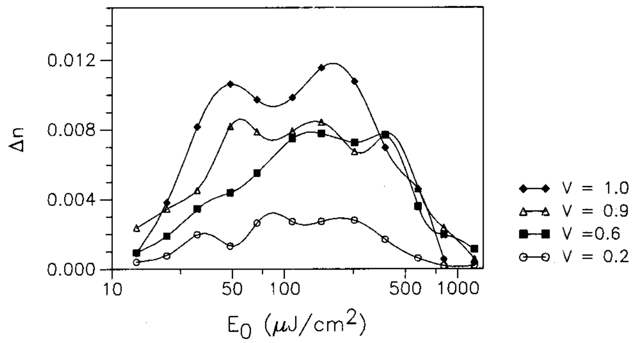

FIG. 4. Measured refractive index modulation vs bias exposure. 100× objective.

eters of the fitted function can be found in Ref. 18.

The microscope used in the measurements of the grating profiles was a Nikon Labophot 2 with a phase-contrast attachment. Each hologram was observed using two phase-contrast microscope objectives. The first one was a 40× (dry) achromate objective (CF DL 40×), of a focal length of 4.4 mm and pupil diameter of 7.53 mm [numerical aperture (NA)=0.65], with the corresponding annular phase plate. The second objective was a CF N Plan DL 100× immersion type one with an NA=1.25 and a focal length of 1.71 mm (in an immersion of cedar wood oil). A green interference filter centred at $\lambda=540$ nm was used. Measurements performed with the 40× (dry) microscope objective yielded the total modulation of the optical path in the gratings, including any possible surface relief, while those performed using the 100× immersion objective yielded only the optical path variations due to the index-of-refraction grating. Each phase contrast micrograph was recorded photographically, using the Micro flex photo micrographic attachment of the Labophot 2 microscope and Kodak HR12 (ISO 100) films. Bias exposure was set to the middle of the quasi-linear range of the $D-\log(E)$ curve of the film. The photomicrographs were scanned in a high-resolution scanner and stored in files.

Examples of phase contrast micrographs and their profiles are shown in Figs. 2 and 3. It can bee seen that even the lowest modulation gratings are discernible.

According to the theory of phase-contrast microscopy, the phase difference in a phase contrast micrograph is

$$\varphi=\frac{\gamma}{2}, \tag{3}$$

where φ is the phase difference in radians and γ is the measured contrast of the phase object.[21]

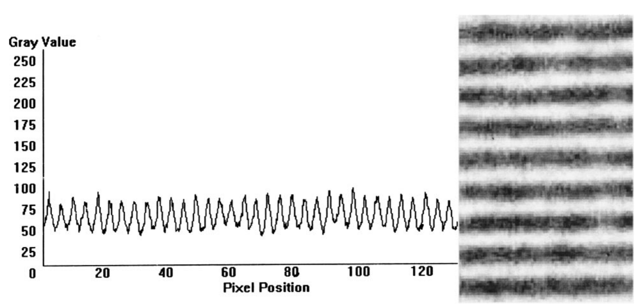

FIG. 3. Phase contrast micrograph of a hologram, point No. 6 of the $V=1.0$ curve on Fig. 1. Microscope objective=100×.

However, especially when fine objects (gratings of high spatial frequency in this case) are studied, one has to take into account the modulation transfer function (MTF) of the microscope objective, too. According to the theory developed by Françon,[6] the contrast of a phase contrast image is influenced by the MTF of the microscope objective in the same way as that of a "normal" amplitude object. Modulation transfer function (MTF) of a diffraction-limited lens is described by,[22]

$$T=2[\arc\cos(K)-K(1-K^2)^{1/2}]/\pi, \tag{4}$$

where T is the MTF value and K is the normalized spatial frequency

$$K=S\lambda f^{\#}, \tag{5}$$

where S is the spatial frequency in lp/mm, λ is the wavelength, and $f^{\#}$ is the ratio of the focal length and the diameter of the lens.

With $\lambda=540$ nm and $S=1200$ lp/mm, in case of the 40× microscope objective we obtain $K_{40}=0.379$ and for the MTF, using Eq. (4): $T_{40}=0.529$. In case of the 100× immersion objective we obtain $K_{100}=0.222$ and $T_{100}=0.720$. So measured values of φ should be divided by these values of K, respectively, to obtain the correct phase differences. Further correction was made for the MTF of the photographic material. Its value for the film and processing used was $M_f=0.417$. The formula for the corrected value of the phase difference is

$$\varphi_{\mathrm{corr}}=\frac{\gamma}{2TM_f}, \tag{6}$$

and hence, the modulation of the optical path becomes

$$\Delta \mathrm{d}=\varphi_{\mathrm{corr}}\lambda/2\pi. \tag{7}$$

Assuming uniform modulation throughout the depth of the holographic emulsion, setting its thickness to $d=5$ μm,[23] we can determine the refractive index modulation, Δn by dividing the Δd values by d. The results obtained with the 100× objective are shown in Fig. 4.

Lin curves of the holograms were calculated from these $\Delta n(E_0,V)$ curves using the Kogelnik formula[24] and were compared to those measured directly. As resolution in the photomicrographs obtained using the 100× immersion objective (NA=1.25) is twice as high as in those obtained using the 40× objective (NA=0.65), only the former results were used for the calculation of the $\sigma(E_0,V)$ curves.

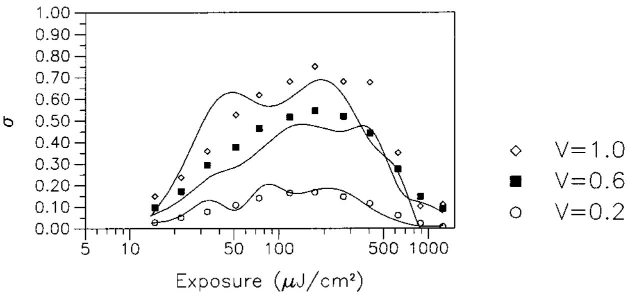

FIG. 5. Measured (symbols) and calculated (lines) values of $\sigma(E_0, V)$. Calculations were performed using the measured Δn values and the Kogelnik formula.

According to coupled wave theory, the square root of the diffraction efficiency of a transmission phase hologram is

$$\sigma(\nu)=\left|\sin\frac{\pi\Delta n d}{\lambda\cos\left[\arc\sin\left(\frac{\nu\lambda}{2}\right)\right]}\right|, \tag{8}$$

where ν is the spatial frequency of the grating, Δn is the modulation of the refractive index, d is the hologram thickness, and λ is the wavelength of the light. Results of the calculations are shown in Fig. 5.

Figure 5 shows a good agreement between the results of the diffraction efficiency measurements and those of the phase contrast microscopy, in spite of the limitations of the latter. It should be emphasized that refractive index modulation was obtained from the phase contrast photomicrographs by fitting a sinusoid to them. Higher-order harmonics[25–27] in the measured grating profiles were also studied by Fourier analysis, and the results will be published soon.

We can conclude that direct observation of the (holographically recorded) phase gratings via phase-contrast microscopy is an adequate method for the quantitative characterization of the gratings even at spatial frequencies as high as 1200 lp/mm and for the determination of the Lin curves of holographic materials.

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