

# Measurement of phase differences between the diffracted orders of deep relief gratings

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Measurement of the phase difference between the 0th and the 1st transmitted diffraction orders of a symmetrical surface-relief grating recorded on a photoresist film is carried out by replacement of the grating in the same setup with which it was recorded. The measurement does not depend on lateral shifts of the replaced grating relative to the interference pattern, on environmental phase perturbations or on the wave-front quality of the interfering beams. The experimental data agree rather well with theoretical results calculated for sinusoidal profiled gratings. © 2003 Optical Society of America

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Surface-relief gratings in both the resonant and the subwavelength domain present interesting polarization properties that can be used to develop new optical elements.<sup>1</sup> Knowledge of both the amplitude and the phase of the diffracted orders is fundamental for the development of such applications. The phases of the diffracted orders in deep surface-relief gratings are not constants, as is the case in shallow gratings or even in volume gratings. These phases depend on the polarization and the wavelength of the incident wave, as well as on the period, depth, and shape of the grating grooves.

There are many reliable numerical methods that can be used to calculate the phase and the amplitude (efficiency) of a diffraction order of a grating.<sup>2</sup> The efficiencies can be easily measured and compared with the theoretical calculation; however, the phase measurements require the use of either ellipsometric or interferometric techniques. The ellipsometric methods are more appropriate for measurement of phase shifts between two orthogonal polarization states of a given diffraction order.<sup>3</sup> For measurements of phase difference between different diffracted orders, the use of interferometry is required. In this case, the phase measurement is very sensitive to the wave-front quality of the interfering waves and to thermal and mechanical perturbations in the experimental setup, increasing the difficulty of measurement.

If the measurement of the interference between the diffracted orders is performed simultaneously in two independent directions, one can compensate for thermal or mechanical fluctuations.<sup>4</sup> This procedure, however, does not compensate for the differences between the interfering wave fronts, introducing errors into the absolute phase measurements. In this Letter we propose and demonstrate an experimental method that permits the measurement of the absolute

phase difference between adjacent diffracted orders of surface-relief gratings.

To explain the method we assume a grating with a period  $\Lambda$ , described by a surface corrugation along the  $x$  axis as depicted in Fig. 1. The two symmetrically incident beams that record the grating form a sinusoidal interference pattern in the  $x$  direction. Any phase change in the incident waves can be represented by a phase  $\psi$  in one of the interfering beams. This phase will shift the interference pattern by the same amount relative to the grating, assuming that the surface-relief grating is placed, at exactly the same location where it was recorded. In this case the intensity of the interference pattern may be represented by

$$I = I_1 + I_2 + 2\sqrt{I_1 I_2} \cos(Kx + \psi), \quad (1)$$

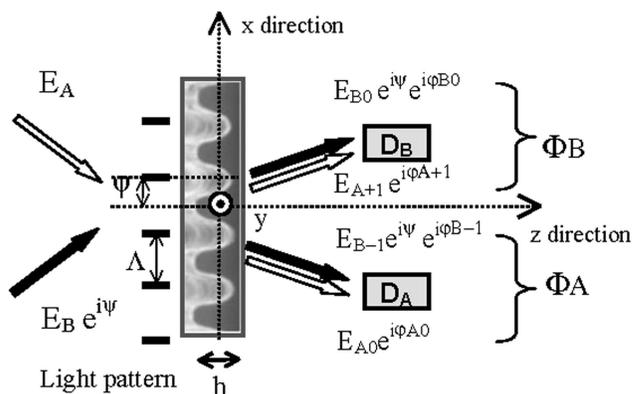


Fig. 1. Scheme of the incident and diffracted waves.  $E_A$  and  $E_B$  are the amplitudes of the incident waves,  $h$  is the grating depth, and  $\Lambda$  is the period.

where  $K = 2\pi/\Lambda$  and  $I_1$  and  $I_2$  are the irradiances of the incident beams represented in Fig. 1 by electric field amplitudes  $E_A$  and  $E_B$ , respectively.

The incident beams are diffracted symmetrically with respect to the grating normal, generating diffracted waves with amplitudes  $E_i$  and phases  $\varphi_i$ . Because the grating has the same period of the interference pattern, the diffracted waves produced by the two incident beams form collinearly propagating pair as shown in Fig. 1. The phase difference  $\Phi_A$  between the 0th transmitted order  $E_{A0}$  produced by the  $E_A$  beam and the  $-1$ st diffracted order  $E_{B-1}$  produced by the  $E_B$  beam is

$$\Phi_A = \psi + \varphi_{B-1} - \varphi_{A0}. \quad (2)$$

Similarly, the phase difference  $\Phi_B$  between the 0th transmitted order  $E_{B0}$  produced by the  $E_B$  beam and the  $+1$ st diffracted order  $E_{A+1}$  produced by the  $E_A$  beam is

$$\Phi_B = \psi + \varphi_{B0} - \varphi_{A+1}. \quad (3)$$

If the grating's surface profile is symmetrical, then  $\varphi_{B0} = \varphi_{A0} = \varphi_0$ . Moreover, if the grating is symmetrically positioned with respect to the two incident plane waves, then  $\varphi_{B-1} = \varphi_{A+1} = \varphi_1$ .

The gratings were recorded in positive photoresist films (AZ1518; Hochst), with a setup described elsewhere.<sup>5</sup> After development in AZ 351 (Hochst) diluted 1:3 in deionized water, each grating was replaced and adjusted in the same angular position where it was recorded. We adjusted each grating by maximizing the period of the Moiré-like fringes,<sup>6</sup> observed in each of the directions of the diffracted beams. The angular adjustment between the grating lines and the interference pattern was made with a precision goniometer that held the grating sample. Figure 2 shows photographs of these Moiré-like patterns for different angular adjustments of the grating. As can be seen from these photographs, the Moiré-like pattern consists of straight fringes whose periods can be made greater than the sample dimensions. This fact occurs because the grating is replaced in the same pattern in which it was recorded, thus yielding the same wave-front distortion, and because, behind the relief, the interfering waves traverse the same optical path. The good amplification obtained in the Moiré-like pattern also demonstrates that the development process does not introduce significant wave-front distortions, because the profile change occurs in a thin layer of the  $z$  axis, whereas the major wave-front information is recorded in the  $x, y$  plane of the photoresist film (Fig. 1).

To perform the measurements we introduce into the holographic setup a phase modulator (a piezoelectric-supported mirror, placed in one of the arms of the interferometer). We may represent this phase modulation by setting  $\psi = \omega t + \psi_0$  in Eqs. (2) and (3). Two identical photodetectors ( $D_A$  and  $D_B$ ) are positioned exactly at the center of sample shadow in the transmitted beams (Fig. 1) so that they collect light from the same region of the sample. Thus, the light intensity projected on photodetector  $D_A$  is

$$I_A = I_{A0} + I_{B-1} + 2\sqrt{I_{A0}I_{B-1}} \times \cos(\omega t + \psi_0 + \varphi_1 - \varphi_0), \quad (4)$$

where  $I_{A0}$  and  $I_{B-1}$  are the intensities of diffracted waves  $E_{A0}$  and  $E_{B-1}$ , respectively. Analogously, photodetector  $D_B$  measures the intensity:

$$I_B = I_{B0} + I_{A+1} + 2\sqrt{I_{B0}I_{A+1}} \times \cos(\omega t + \psi_0 - \varphi_1 + \varphi_0). \quad (5)$$

Because the cosine is an even function, the result does not depend on the signs of  $\Phi_A$  and  $\Phi_B$ . Thus, the phase difference  $\Delta$  between the beams collected by the two photodetectors is

$$\Delta = \pm 2(\varphi_1 - \varphi_0), \quad (6)$$

which is twice the phase difference between the first and the 0th transmitted orders,  $\varphi_1 - \varphi_0$ . Note that this result is independent of the phase  $\psi_0$  that represents phase perturbations in the interferometer or lateral shifts of the grating relative to the interference pattern.

We recorded gratings with the same period ( $\Lambda = 0.8 \mu\text{m}$ ) but different depths by changing the exposure energy and the development time. This procedure, however, led to a set of gratings whose profiles can deviate substantially from the sinusoidal form.<sup>7</sup> We measured the phase differences  $\Delta$  [Eq. (6)] by inputting the signals from the two photodetectors [proportional to the intensities  $I_A$  and  $I_B$  in Eqs. (4) and (5), respectively] in the two orthogonal axes of an oscilloscope. Figure 3 shows the ellipses, corresponding to the measurement of three samples, in the inset of each scanning electron microscope sample cross-section photograph. From the change of the ellipses one can observe a large phase variation in the samples.

Figure 4 shows the values of the phase difference between the 1st and the 0th diffracted orders ( $\varphi_1 - \varphi_0$ ) measured for 13 labeled samples with equal period and different depths. The horizontal error bars were evaluated from the direct grating depth measurement in the scanning electron microscope cross section of the central region of the samples. The variations of grating depth along the sample measured area were not considered. We estimated the phase error bars by adding the direct errors of the ellipse phase measurement with the error that was due to the noninfinity Moiré-like period. This last error was estimated by the relation between the diameter of the active area of the detector (averaged measured phase)



Fig. 2. Photographs of the Moiré-like pattern formed in each direction of the detected beams. The left-hand image corresponds to the worst angular adjustment; the right, to the best.

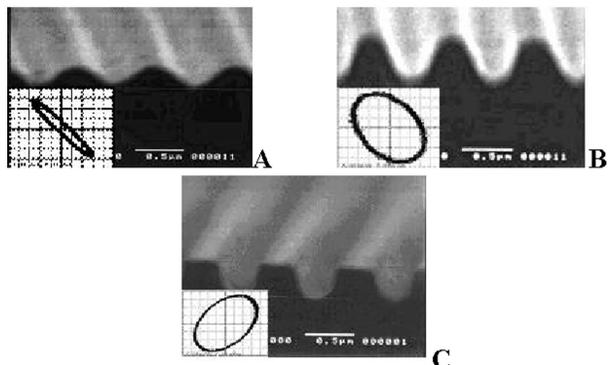


Fig. 3. Scanning electron microscope photographs of the cross section of three samples with different depths and profiles: A, 12; B, 11; and C, 1. The ellipses formed in the phase difference measurements are shown in the inset of each photograph.

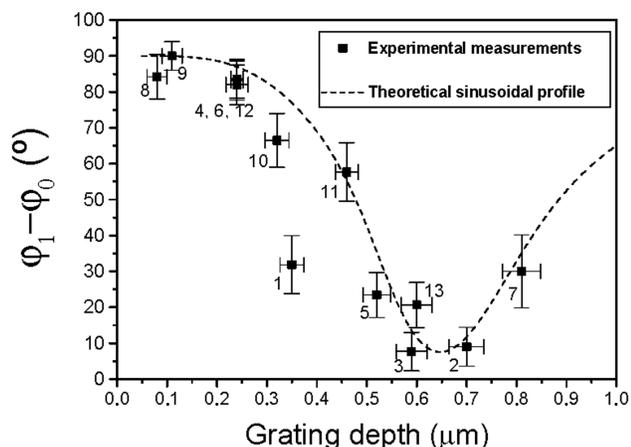


Fig. 4. Experimental  $(\phi_1 - \phi_0)$  measurements for 13 samples with different depths and profiles. The numbers of the samples are indicated beside the squares.

and the Moiré-like period, which corresponds to a phase variation of  $2\pi$ .

The measured  $(\phi_1 - \phi_0)$  values are quite repetitive for different replacements of the same grating, demonstrating that the measurement is independent of the sample positioning. In addition, the samples 4, 6, and 12 in Fig. 4, which have the same depth ( $\approx 0.25 \mu\text{m}$ ), yielded the same  $(\phi_1 - \phi_0)$  measured values. As the substrates of the samples are different, this fact demonstrates that the method compensates for the wave-front distortion that is due to the different optical path behind the grating.

Besides the dispersion, the  $(\phi_1 - \phi_0)$  measurements demonstrate well-defined behavior: Starting from the value  $\pi/2$ , the measurements decrease as a function of the grating depth, reaching a minimum at a depth of 0.6 and 0.7  $\mu\text{m}$ . For comparison, in Fig. 4 we also plot the theoretical  $(\phi_1 - \phi_0)$  curve as a function of the grating depth for a perfect sinusoidal grating with the same period ( $\Lambda = 0.8 \mu\text{m}$ ), wavelength ( $\lambda = 0.4579 \mu\text{m}$ ), polarization (TE), and incident angle used in our experiments. The curve was computed with the coordinate transformation method (the C method).<sup>8</sup> Note that for this grating

period and this condition of incidence, called first-order Littrow mounting,<sup>2</sup> the phase difference at the wavelength  $0.4579 \mu\text{m}$  deviates significantly from  $90^\circ$  for groove depth-to-period ratios greater than  $1/4$ , and the curve's minimum is at  $0.65 \mu\text{m}$ . Although the  $(\phi_1 - \phi_0)$  curve as a function of the grating depth should depend on the grating profile, at small depths this phase should start from  $\pi/2$ , independently of the grating profile, as expected for shallow phase gratings, from scalar theory.

As can be seen in Fig. 4, the  $(\phi_1 - \phi_0)$  measurements follow the behavior expected for sinusoidal gratings. The agreement is particularly good for the samples with sinusoidal profile [samples 12 and 11 Figs. 3(a) and 3(b), respectively], whereas for samples 1 [Fig. 3(c)] and 10, which are nonsinusoidal, the measured  $(\phi_1 - \phi_0)$  values depart from the theoretical curve. For the deeper samples (2, 3, 5, and 13), whose profiles are also nonsinusoidal, the departure from the theoretical curve is smaller, because they are in a part of the curve with a smaller derivative (less sensitive to the depth). For the shallow samples, the discrepancies come essentially from experimental errors in both phase and grating depth measurements.

In conclusion, the results demonstrate the ability of the proposed method to measure the absolute phase difference between the diffracted orders of relief gratings. The method can be applied for gratings with different relief profiles and thus constitutes a powerful tool for fundamental studies of the phases between the diffracted orders and for checking the results predicted by diffraction theories. The main requirement for the applicability of the method is good angular alignment of the sample with the interference pattern. The homogeneity in the grating depth along the sample is also very important, because different depths introduce different phases into the diffracted wave fronts, thus generating a corresponding phase deformation in the Moiré-like pattern that can introduce errors into the measurement.

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