44

Technological problems in holographic recording of plane gratings

Evgueny K. Popov
Lyubomir V. Tsonev
Maria L. Sabeva
Bulgarian Academy of Sciences
Institute of Solid State Physics
72 Trakia Boulevard
1784 Sofia, Bulgaria

Abstract. Solutions to several problems in relief grating holographic fabrication are presented. (1) The requirements for high-spectral selectivity and for a low stray light level are contradictory, but this problem can be solved by a compromise in the recording arrangement. (2) A real-time groove-depth control is proposed that ensures the fast and effective determination of the accurate exposing and developing conditions. (3) The necessity of baking the photoresistive grating before its metalization is clearly demonstrated by experiment.

Subject terms: light diffraction; relief gratings; holographic recording.

Optical Engineering 31(10), 2168–2173 (October 1992).

1 Introduction

A strong belief exists that holographic recording of relief diffraction gratings is a high-tech process with predominant advantages over the classical mechanical ruling. Indeed, developing a ruling engine has always been considered (and really is) a work of art. In comparison with almost two centuries of pushing optomechanics through thick and thin the history of holographic recording seems to be child's play—standard holographic equipment and a good quality laser is all that's needed.[1–3]

In reality, it is not so easy. What made gratings cheaper and easily available was not their optical recording but rather the recent techniques for replication.[4] Manufacturing of a holographically recorded master of true spectrometric quality is achieved almost as rarely as a similar-ruled piece. Most readers are likely to be acquainted with the advantages of optical recording—absence of ghosts, higher SNR, a faster production process, etc. However, rarely is it pointed out that a number of specific problems exist in optical recording that can readily reduce these advantages to zero. For example, a stray gleam of a laser beam by or through some surface that is planned to be nonreflective, untransparent, and holeless could result in ghosts as real as any diffraction order, because such gleams act as additional light sources coherent to the main recording beams. Unfortunately, such a negative experience usually lies outside the scope of positive knowledge. Although, in our opinion, this experience is much more useful for technological development than the common general advice. The situation is quite similar to computer programming—"debugging" (searching for mistakes and reasons for their appearance) is a much more time- and patience-consuming process than the programming itself.

The aim of our paper is to present some difficulties in the technological chain of processes for holographic recording of metalized relief diffraction gratings. Where possible we attempted to overcome these difficulties. We devote our attention to three main stages of optical recording: (1) design of the recording setup, (2) real-time groove depth measurements, and (3) reflection layer coating. Our main criterion has always been the quality of the final product as expressed in its diffraction efficiency. Of course, errors could arise at every step of the process, starting from substrate polishing and finishing with leaving footprints on the grating surface. We mention the following case as an example: simply covering the aluminum grating with a dielectric layer to protect it from oxidizing and dirt can lead unwittingly to the proper conditions for the appearance of new strong anomalies, caused by guided mode excitation in such a layer.[5,6]

Paper 07111 received Nov. 8, 1991; revised manuscript received Jan. 24, 1992; accepted for publication Jan. 25, 1992.

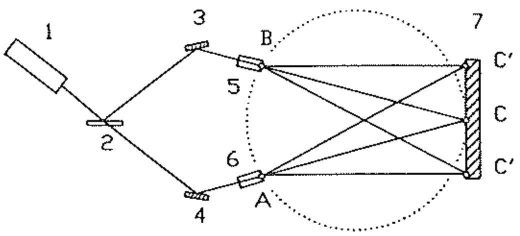


Fig. 1 Plane grating recording scheme using two spatially filtered, but not collimated beams. 1—laser, 2—beam splitter, 3,4—mirrors, 5,6—spatial filters, 7—grating blank. Dotted line—circle defined by the points A, B, and C.

In what follows we try to distinguish between the influence of the three previously mentioned steps, although their improper realization has almost the same effect on grating response—the diminishing of real efficiency in comparison to the ideally predicted and expected value. For example, violation of either the recording or aluminum deposition conditions can reduce the measured efficiencies even for optimally chosen groove depth, but through different physical mechanisms: an increase of the diffuse scattering (Sec. 2) or of absorption (Sec. 4), respectively.

2 Recording Set Arrangement

It is a well-known fact that to produce high-quality plane diffraction gratings by means of holographic recording two [(1) spatially filtered and (2) collimated] coherent monochromatic beams interfering under specified angles of incidence are needed in a photoresist layer that is sensitive to the wavelength used. Unfortunately, neither of the above requirements can be fulfilled precisely without a violation of the other.

If we prefer to attack first parasitic stray light, we must use spatially filtered recording beams without subsequent collimation, i.e., the simplest scheme (Fig. 1). We can check the scattering level, e.g., illuminating the grating by a narrow laser beam and examining the diffracted one. The cross sections of the incident and of the diffracted beams are shown in Figs. 2(a) and 2(b), respectively. The diffracted beam [Fig. 2(b)] does not reach the camera to prevent the film from overexposing; therefore, the location of the main beam is indicated only by a black spot in the center and the remainder of the image is the stray light halo. The scattering is very low ($\leq 1\%$) and is connected with the quality (smoothness) of the photoresist grooves and of the cladding aluminum layer.

The low stray light level of such a grating is, unfortunately, combined with a slow change of the groove period over the exposed area, which diminishes the resolving power: The period change affects negatively the imaging properties of a parallel beam spectrograph containing such a grating. The local period is determined by the angle $\angle$ACB in the center and by $\angle$AC'B at the periphery, where $\angle$AC'B $<$ $\angle$ACB caused by the beam divergence (Fig. 1). If the period varies more than $\varepsilon = \Delta d/d_0$ (d_0 is the period in the center of the grating and Δd is the maximum period variation), relative spectral selectivity better than $\varepsilon = \Delta\lambda/\lambda$ can hardly be achieved with using the entire grating area. Such a defect is clearly manifested when a sufficiently wide well-collimated beam reaches the grating: After the diffraction, the beam loses its collimation—its cross section is no longer a rectangle similar to the grating blank itself. The cross section is deformed into a trapezoid, shown in Fig. 3(a). Therefore, if one tries to focus such a beam with a standard spherical mirror or lens, one can never obtain a point; the real focal spot [Fig. 3(b)] makes it impossible to use such grating in a parallel beam spectral device. In summary, gratings recorded by filtered but divergent beams have low scattering, but also low resolution.

If we prefer, on the other hand, to achieve high-spectral resolution, we can record the grating using well-collimated beams, produced by off-axis parabolic mirrors situated after the spatial filters (Fig. 4). In this case, the grooves are strongly straight, parallel, and equidistant over the entire area. Illuminating the grating with a wide-collimated beam, a well-collimated diffracted beam is also obtained with a cross section that exactly reproduces the blank form [Fig. 3(c)]. As can be expected, focusing such a beam by a concave mirror results in a small pointlike focal spot [Fig. 3(d)], ensuring good spectral resolution in a parallel beam device.

Unfortunately, collimated beams no longer have the properties of spatially filtered beams; therefore, light scattering from the grating so recorded is greatly increased: A narrow incident laser beam with a cross section depicted in Fig. 2(a) is transformed after diffraction in a narrow central beam surrounded by a relatively bright stray light conical halo with a cross section shown in Fig. 2(c). This phenomenon can reduce diffraction efficiency (usually measured with a small-area detector at the center of the narrow laser beam) by 20 to 30%. The divergence angle of this cone is determined by the ratio between the concave mirror diameter and the distance between the mirror and grating. Each imperfection on the mirror surface acts as a microscopic scattering center; the local intensity of the scattered light decreases as L^{-2}, where L is the distance measured from the mirror surface. Because of the absence of spatial filtering, all of these secondary waves reach the grating blank and are registered on the photoresistive layer together with the two main collimated beams. In other words, because of their scattering effect, the mirrors are holographically recorded in the grating layer and, therefore, their images are also holographically reproduced every time the grating is illuminated. One can observe, e.g., in Fig. 2(c), the bright peripheral arcs caused by the significant scattering from the faces of the mirror blanks.

The contradiction between spectral resolution and stray light level can be solved, e.g., by a compromise—a recording scheme of the type shown in Fig. 4 can be used, but with a larger distance L between the mirrors and grating blank. Doubling the distance causes a stray-light reduction of four times, as illustrated in Fig. 2(d). Such techniques, of course, can be recommended only if a sufficiently large holographic table is available. The resolving properties of such an improved grating, i.e., its imaging characteristics under parallel beam illumination and diffracted light focusing, are the same as those previously shown in Figs. 3(c) and 3(d).

3 Real-time Groove Depth Evaluation

The predominant case of holographic recording uses a common symmetric scheme. Without precautions, the profile of

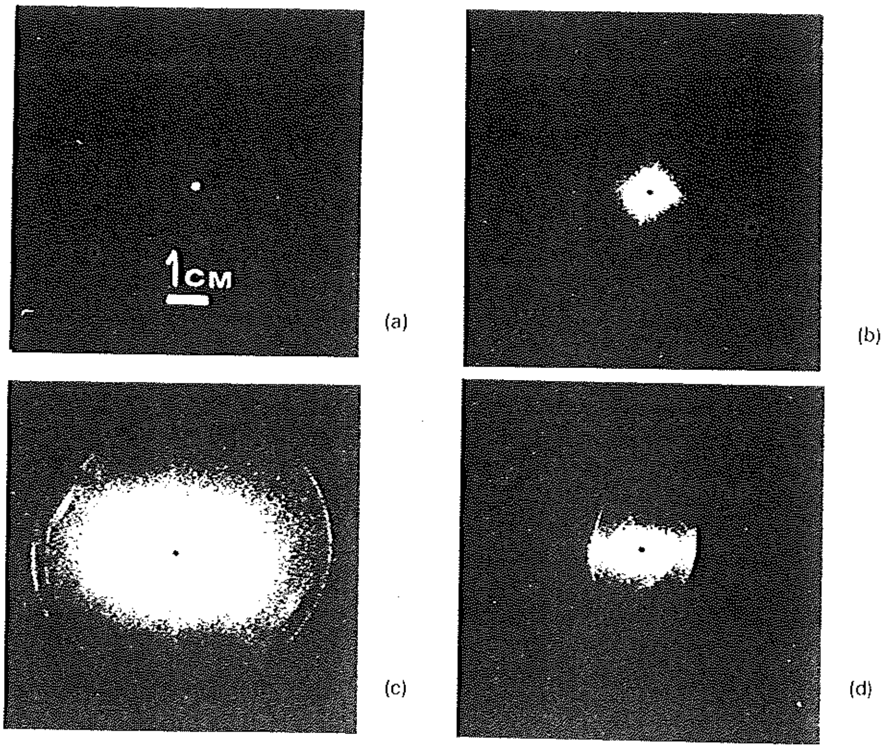


Fig. 2 Laser beam cross-section photographs: (a) incident beam; (b), (c), and (d) show a stray light halo around the diffracted beam. (b) very low, (c) very high, and (d) properly minimized scattering. In case (b) the grating is recorded with two filtered but not collimated beams. In cases (c) and (d) the grating is recorded with two collimated but thereafter not filtered beams. Camera position remains fixed in all the cases.

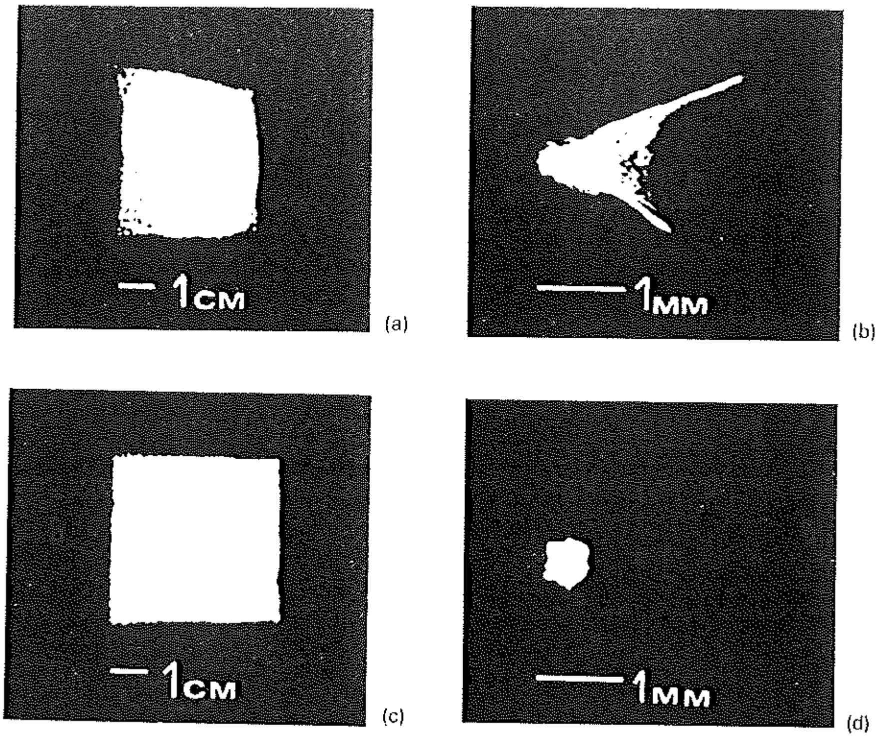


Fig. 3 Diffracted beam cross-section photographs when a rectangular grating is illuminated by an expanded collimated beam entirely covering its area: (a) and (c) without any optical treatment of the diffracted beam and (b) and (d) after focusing the diffracted beam with a spherical mirror or lens. (a) and (b) the grating has been recorded by spatially filtered but not collimated beams and (c) and (d) the grating has been recorded by collimated but not spatially filtered beams. Camera position remains fixed in all the cases.

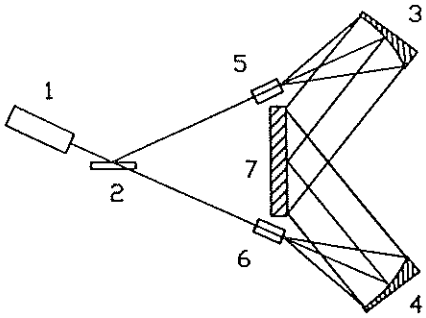
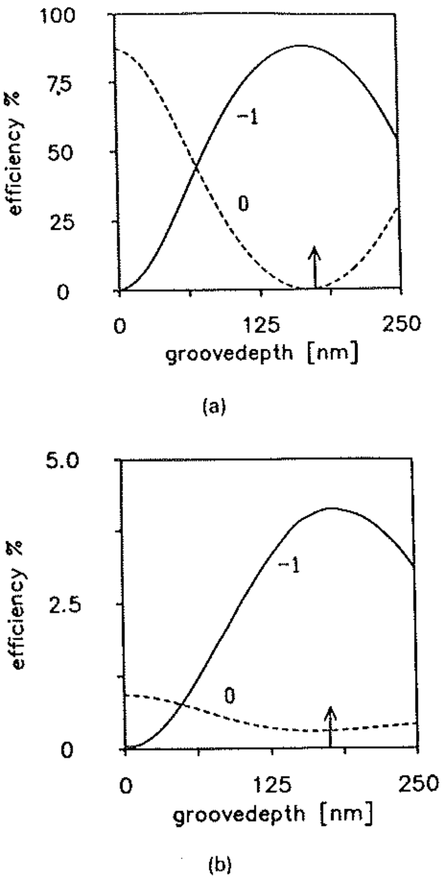


Fig. 4 Plane grating recording scheme using two collimated but thereafter not spatially filtered beams. Numbers are as in Fig. 1.

the grooves recorded typically has a quasi-sinusoidal form.[7,8] Taking into account that the period is inevitably fixed by the primary choice of interference angle and recording wavelength, the only parameter that determines the resulting grating characteristics (e.g., spectral efficiency) is the groove depth. Groove shape is formed during the development of the exposed photoresist and groove depth h depends on the exposure time and intensity, developer concentration, temperature, and developing time. These dependencies were investigated thoroughly and, in general, a desired groove-depth value could be obtained by a proper choice of developing time with other conditions fixed. However, in practice, deviations in the exposing and developing process from the ideal often alter results unpredictably. A typical difficulty is small differences between the intensities of the two recording beams.

Usually the information about any such fault in the recording process is obtained only at the end of the entire technological chain—after aluminization of the grating. While point defects and strong nonhomogeneity of the grating along the blank can be easily observed before metal coating, relatively precise determination of groove-depth correctness is usually accomplished by measuring the final product properties (metallic grating efficiency). This final test is not too useful for feedback correction of the exposure and developing processes. To obtain information about the groove-depth values immediately after developing, washing, and blank drying occurs is much more convenient. If we know the theoretical efficiencies of the aluminum and of the photoresist grating as a function of the groove depth at fixed identical conditions (a sinusoidal groove shape is assumed as a satisfactory approximation to the real one), we should be able to control the groove depth not by measuring the efficiency of the final metal grating, but by measuring that of the photoresist grating. Such theoretical data can be calculated with the help of any of the rigorous theoretical methods.[9]

Theoretical groove-depth dependence of 0th and -1st reflection order efficiencies for TM-polarized light are shown in Figs. 5(a) and 5(b) for aluminum and dielectric grating without scattering losses. The period is $d = 0.5$ μm, the wavelength is $\lambda = 0.6328$ μm, and the angle of incidence corresponds to 2-deg angular deviation from the Littrow mount. Our goal was to produce a dye-laser diffraction grating with substantially high -1st-order diffraction efficiency in TM polarization in the red spectral region.[10–12] According to Fig. 5(a), such an aluminized grating must have a groove depth $h = 180$ nm (modulation 36%) and will possess 0% 0th-order and 88% -1st-order efficiency at $\lambda = 0.6328$ μm.



(a)

(b)

Fig. 5 Theoretical TM-diffraction efficiency of 0th and -1st reflection orders as a function of groove depth at a 0.6328-μm wavelength and for sinusoidal groove shape: (a) for an aluminum grating and (b) for a photoresistive grating.

After a proper preliminary choice of exposure intensity and time, we identically recorded a few (four are usually enough) photoresist-coated blanks. Three of them were relatively small subsidiary samples. They were developed for successively increasing time intervals. The efficiency was measured in reflection for each sample. (Note that it is not convenient to use transmitted orders.) The proper developing time was determined by a simple linear interpolation of dielectric efficiency data according to Fig. 5(b) instead of Fig. 5(a). The desired groove depth corresponds to 4.1% -1st-order and 0.3% 0th-order efficiency of the photoresistive grating at $\lambda = 0.6328$ μm as indicated with an arrow in Fig. 5(b). Following this selection procedure, the main (original) sample was developed. It is important to note here that if the grating is to be used as a replication master, a proper preliminary correction of the groove depth should be introduced to compensate for the epoxy shrinkage during curing.[3,4]

Of course, it is possible to work with a single blank using multiple developing and measurement, but it becomes riskier with surface defects or dirt on its photoresist surface.

We have produced two identically recorded original samples, I and II, in this manner, differing only in the alumin-

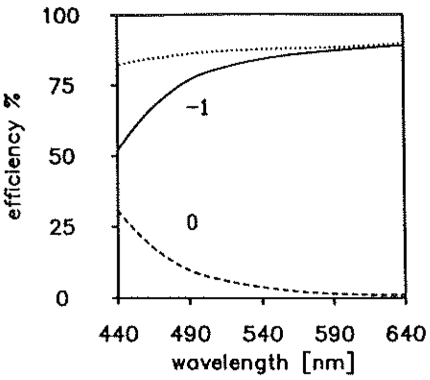


Fig. 6 Theoretical spectral efficiency behavior of aluminum coated grating in 0th and − 1st TM reflection order (sinusoidal groove shape, 0.5-μm period, 180-nm groove depth). Dashed line—zeroth order, continuous line—first order and dotted line—total efficiency.

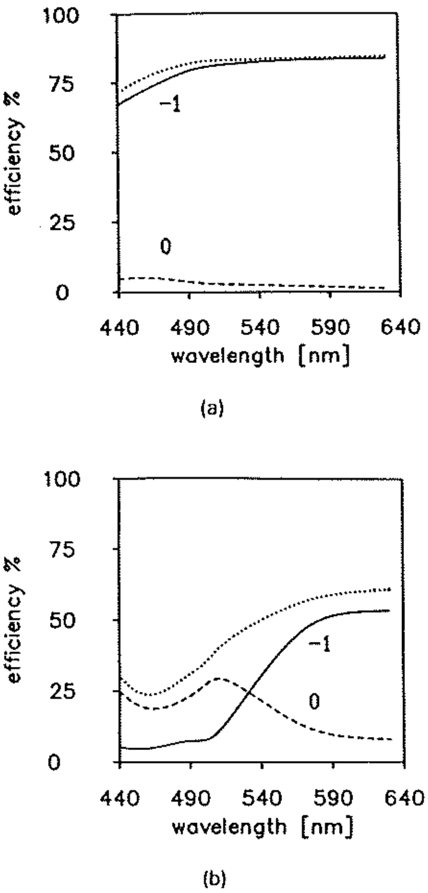


Fig. 7 Experimental spectral behavior of 0th and − 1st TM reflection order efficiencies (other data and line types are as in Fig. 6): (a) for grating I (baked before metalization) and (b) for grating II (not baked before metalization).

ization technique. Their spectral behavior is presented and discussed in detail in the next section.

4 Aluminum Grating Efficiency

The theoretical spectral TM-efficiency behavior of a diffraction grating with sinusoidal-shaped grooves coated with aluminum is shown in Fig. 6. Complete absence of scattered light loss is assumed here—we suppose that the metal layer is ideally smooth, the recording beams are collimated, and the off-axis mirrors are far enough from the grating blank. A groove-depth value of 180 nm is chosen to provide maximum − 1st-order efficiency for TM-polarized light. Figures 7(a) and 7(b) present the experimental spectral results for the two identically recorded and developed samples I and II mentioned in the previous section. Measurements were carried out using a set of lasers: He-Cd, Ar^+, He-Ne, Kr^+, and a dye laser.

Only one technological difference exists between gratings I and II. The first sample I [Fig. 7(a)] was baked for 6 h at 120°C before metalization, whereas the second sample II [Fig. 7(b)] was coated with aluminum immediately after developing and dried at room temperature. Their significantly different spectral behavior can be explained as follows: The unbaked photoresist layer exhales vapors of water and of developer solvent during the coating process. These vapors damage the aluminum layer. Therefore, the metalized sample II has a slightly yellowish-brown color in reflection, easily understood in light of Fig. 7(b); sample II exhibits a strong absorption in the blue region. It should be pointed out again that both gratings I and II were made under identical geometrical, exposure, and developing conditions chosen according to Sec. 2, to minimize the scattering losses. However, only the proper aluminization technique applied on sample I ensures the desired and predicted high efficiency, as it can be proven by a simple comparison between the total diffraction efficiency data presented in Figs. 6, 7(a), and 7(b).

5 Conclusions

Some difficulties in the fabrication process of planar relief holographic metalized gratings are presented, their origins are discussed, and solutions to overcome them are proposed. The first problem is to find an experimental balance between the requirements for high-spectral selectivity (i.e., ideally straight and equidistant grooves, generated by two collimated but, therefore, spatially unfiltered laser beams) and for low stray light level (i.e., ideally smooth grooves, formed by two spatially well-filtered but, therefore, uncollimated laser beams). We suggest here to move the collimating mirrors as far away as possible from the grating blank.

The second problem is how to control effectively the groove depth (i.e., exposing and developing conditions) to achieve its optimum value with a high degree of certainty. Our solution consists of proper diffraction efficiency measurements on the photoresistive grating before its metalization. Such a procedure is much faster, more convenient for feedback, and cheaper than measurements on the final metalized sample.

The third problem is the quality of the aluminum coating. We find here that a prebaking of the photoresistive grating before metalization is essential to prevent the strong cladding absorption in the blue spectral region.

Acknowledgments

The authors thank Dr. G. Zartov for high-quality aluminum deposition and the reviewers for improving the manuscript.

References

1. E. G. Loewen, "Current and future grating technology," *Vistas in Astronomy* **29**(2), 223–236 (1986).
2. E. G. Loewen, R. S. Wiley, and G. Portas, "Large diffraction grating ruling engine with nanometer digital control system," in *Application and Theory of Periodic Structures, Diffraction Gratings and Moire Phenomena III*, J. M. Lerner, Ed., *Proc. SPIE* **815**, 88–95 (1988).
3. E. G. Loewen, "Diffraction gratings," *Opt. Eng.* **15**(5), 446–450 (1976).
4. H. M. Weissman, "Epoxy replication of optics," *Opt. Eng.* **15**(5), 435–441 (1976).
5. L. Mashev and E. Popov, "Diffraction efficiency anomalies of multicoated dielectric gratings," *Opt. Commun.* **51**(3), 131–136 (1984).
6. L. Mashev and E. Popov, "Zero order anomaly of dielectric coated gratings," *Opt. Commun.* 55(6), 377–380 (1985).
7. L. Mashev and S. Tonchev, "Formation of holographic diffraction gratings in photoresist," *Appl. Phys.* **A26**(3), 143–149 (1981).
8. S. Lindau, "The groove profile formation of holographic gratings," *Opt. Acta* **29**(10), 1371–1381 (1982).
9. J. Chandezon, T. Dupuis, and G. Cornet, "Multicoated gratings: a differential formalism applicable in the entire optical region," *J. Opt. Soc. Am.* **72**(7), 839–846 (1982).
10. M. G. Littman, "Single-mode operation of grazing-incidence pulsed dye laser," *Opt. Lett.* 3(4), 138–140 (1978).
11. F. J. Duarte and J. A. Piper, "Comparison of prism-expander and grazing-incidence grating cavities for copper laser pumped dye lasers," *Appl. Opt.* **21**(15), 2782–2786 (1982).
12. M. N. Nenchev, "Cavity configuration in a dye laser for dispersion on the two output beams," *Opt. Commun.* **50**(1), 36–40 (1984).

Evgueny K. Popov graduated from the Physical Faculty of Sofia University in 1982. Since then he has been working at the Institute of Solid State Physics on the theoretical, experimental, and technological problems of relief diffraction gratings. He obtained his PhD in 1988 and ScD in 1991. Since 1991, he became an associate professor. Popov is a member of the Optical Society of America and of the Union of Scientists in Bulgaria.

Lyubomir V. Tsonev graduated from the Sofia University in 1978 and began to work at the Institute of Solid State Physics, examining planar optical waveguides in glass and in $LiNbO_3$. In 1986 Tsonev received a PhD in integrated optics. Since 1988 he has been involved in investigations on planar and concave holographic diffraction gratings. Tsonev is a member of the Union of Scientists in Bulgaria and of the Bulgarian Physical Society.

Maria L. Sabeva graduated from the Moscow State University in 1971. In 1982 she completed her PhD thesis there on "Coherent anti-stokes raman spectroscopy and anisotropic rayleigh scattering in nematic liquid crystals." From 1983 to 1989 she was a research scientist at the Institute of Electronics. Since 1989 she has been at the Institute of Solid State Physics in Sofia. Her main area of interest is the linear and nonlinear interaction of electromagnetic waves with relief gratings. Sabeva is a member of the Bulgarian Physical Society.