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## EXPERIMENTAL SPECTRAL ELLIPSOMETRY OF 1D DEEP LAMELLAR GRATINGS

### EXPERIMENTÁLNÍ SPEKTRÁLNÍ ELIPSOMETRIE JEDNODIMENZIONÁLNÍCH HLUBOKÝCH LAMELÁRNÍCH MŘÍŽEK

#### Abstract

The phase modulated spectroscopic ellipsometry (PMSE) as the excellent experimental technique for the specification of optical and geometrical parameters of thin films and multilayer dielectric coatings has been applied for the inspection of one-dimensional deep gratings. The experiments have been realized at the incidence angles from 48 to 75 degrees (depending on the kind of measurement) for the zero reflected order in the spectral region from 270 to 700 nanometers. The lamellar gratings have been realized by etching method on the common SiO<sub>2</sub> substrate. The main attention has been concentrated to the experimental study of the influence of the groove breadth, mutual position of incidence plane and grating geometry, and incidence angles on ellipsometric angle dispersion of diffracted light.

#### Abstrakt

Fázově modulovaná spektrální elipsometrie (PMSE) jako vynikající experimentální technika pro určování optických a geometrických parametrů tenkých vrstev a dielektrických povrchů tvořených multivrstvami byla využita pro výzkum jednorozměrných hlubokých mřížek. Experimenty byly prováděny pro úhly dopadu od 48 do 75 stupňů (v závislosti na druhu měření) pro nulový reflexní řád ve spektrální oblasti 270 až 700 nanometrů. Lamelární mřížky byly připraveny leptací metodou na společné podložce z oxidu křemíku. Hlavní pozornost byla soustředěna na experimentální studium vlivu šířky vrypu, vzájemné polohy roviny dopadu světla a mřížkové geometrie a úhlů dopadu světla na disperzi elipsometrických úhlů difraktovaného světla.

**Key words:** spectral ellipsometry, gratings, light diffraction.

#### Introduction

The phase modulated spectroscopic ellipsometry (PMSE) is the excellent experimental technique for the specification of optical and geometrical parameters of thin films and multilayer dielectric coatings. This technique has been applied ex situ and in situ for the growth control of transparent films with varying composition [1]. The recent advances in PMSE instrumentation are summarized in [2].

The lamellar gratings are attractive in optoelectronics, spintronics, visible and infrared optics etc. Scattering of TE-polarized plane waves by a finite number of rectangular grooves in perfectly conducting films and giant resonant enhancement of the electric field within the grooves is reported [3]. The resonant grating reflection filters operating at normal incidence are investigated. Multimode structures are shown to exhibit broader angular selectivities and narrower spectral linewidths than those of single-mode structures [4]. Anisotropic etching of v-shaped grooves in (100) crystalline silicon has been proposed as a method to realize

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transmission-type Fourier-plane multiple beam splitter gratings for infrared wavelengths [5]. The asymmetric beam deflection by doubly grooved binary gratings was demonstrated theoretically and experimentally [6]. It has been shown that the optical response of regularly arranged noble metal wires with nanoscopic cross sections (nanowire gratings) strongly depends on the polarization direction of the incident light [7].

In the present paper the spectral ellipsometry has been applied for the specification of diffractive properties of deep SiO<sub>2</sub> periodical structures for light spectrum from 270 to 700 nanometers.

The attention has been concentrated on the experimental study of the influence of the groove breadth, mutual position of incidence plane and grating geometry, and incidence angles on ellipsometric angle dispersion of diffracted light.

### Experimental arrangement

The experiments were performed using computer controlled four zone null ellipsometer with polarizer-sample-compensator-analyzer (PSCA) configuration in the spectral region from 270 to 700 nanometers (Fig. 1). A Xe-Hg lamp was used as a light source. A 10 cm double-grating monochromator combined with a photomultiplier was employed as a null detector. Rochon quartz prism polarizer and analyzer were rotated by stepping motors to adjust a minimal intensity. A four total reflection-type Fresnel rhomb made from two fused silica rods was used as an achromatic quarter wave retarder. The four-zone averaging for two compensator azimuth angles  $\pm 45^\circ$  gives high precision measurements insensitive to the compensator, polarizer and analyzer azimuth angle errors and imperfections. Absence of systematic errors is essential for a reasonable determination of the unknown parameters. The experiments have been realized at the incidence angles from 48 to 75 degrees (dependent on the kind of measurement) for the zero reflected order. The plane of incidence was adjusted to be parallel or diagonal to the lamellar geometry.

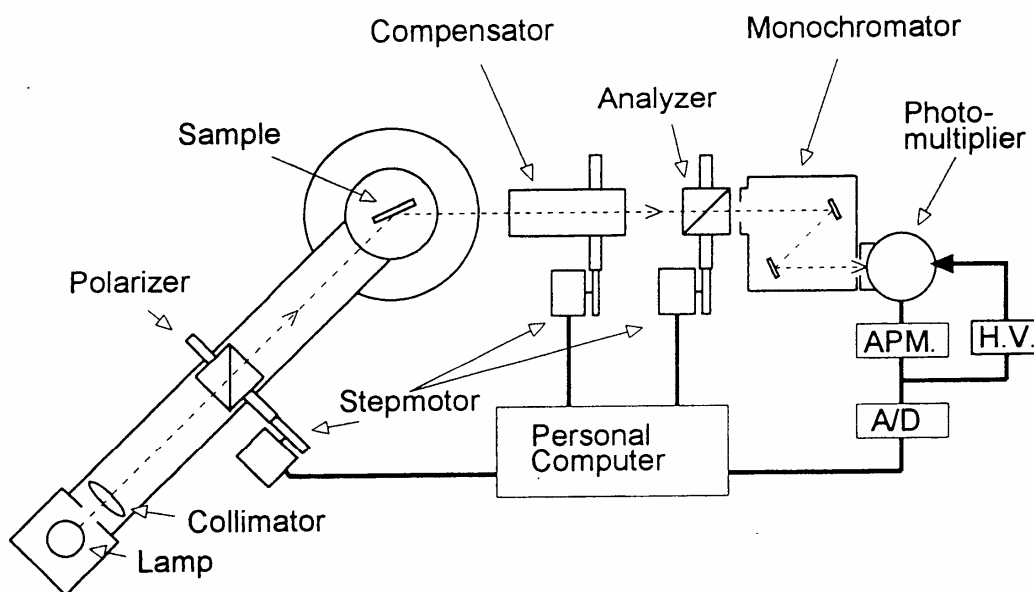


Fig. 1. Experimental arrangement of spectral ellipsometry

### Samples

The set of 5 to 5 mm lamellar gratings has been prepared by etching method on the common SiO<sub>2</sub> substrate. The periodicity  $d$  (see Fig. 2) and the depth  $h$  of all samples are the same: 260 nm and 525 nm, respectively. The groove breadths ( $s$ ) have been specified by Electron Microscopy (EM) method with the following results: A sample – 92 nm, B sample – 96 nm, C sample – 114 nm, D sample – 153 nm, E sample – 171 nm.

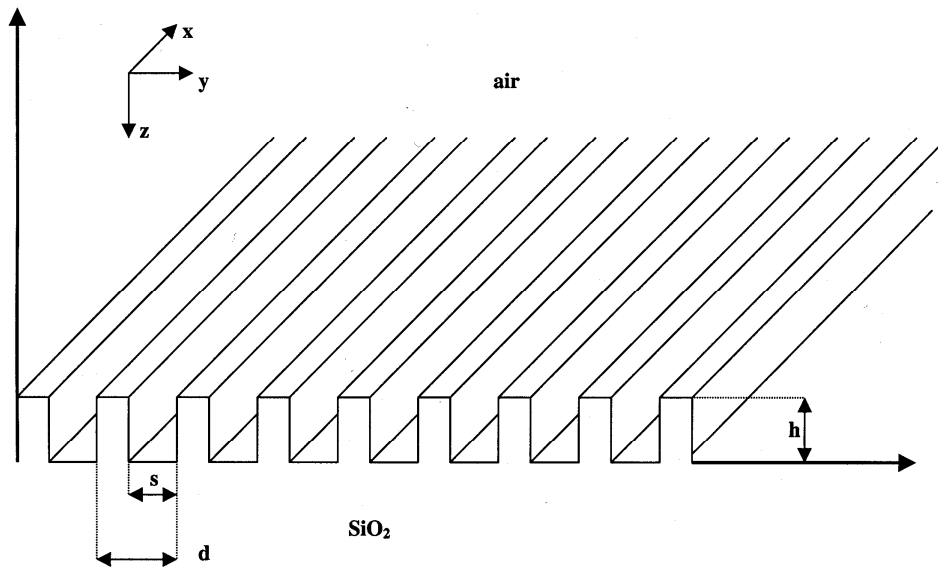


Fig. 2. Geometry of lamellar gratings

### Results and discussion

The experimental results are demonstrated in the collection of figures. The influences of the mutual geometrical position of incidence optical plane and grating grooves, the incidence angles of light beam, and groove breadths are inspected by experimental way in detail.

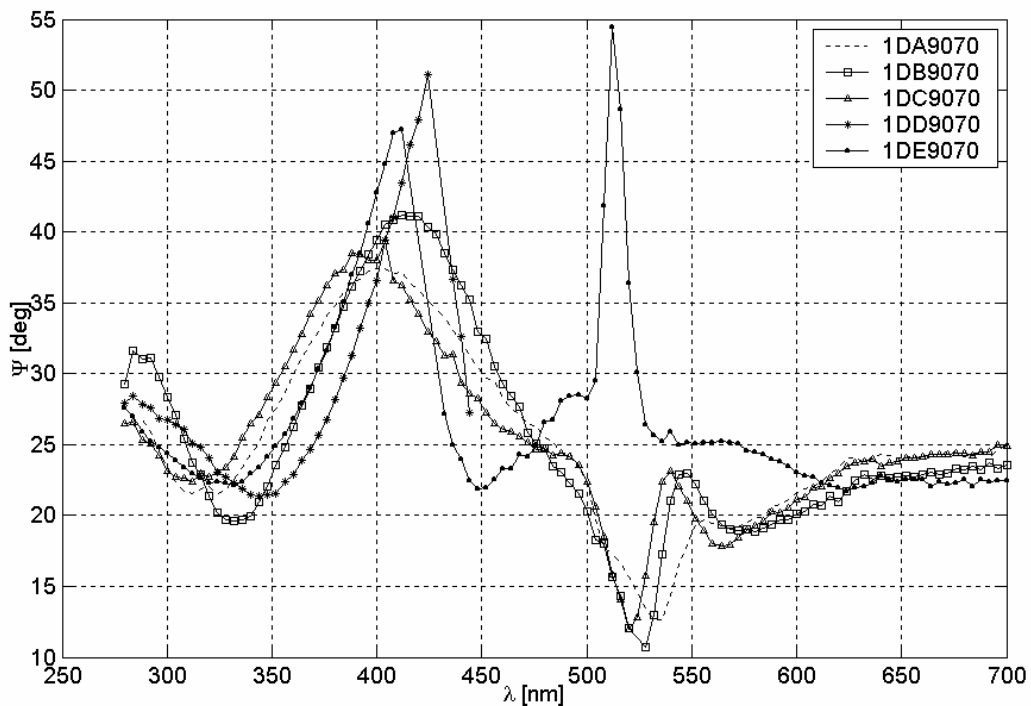
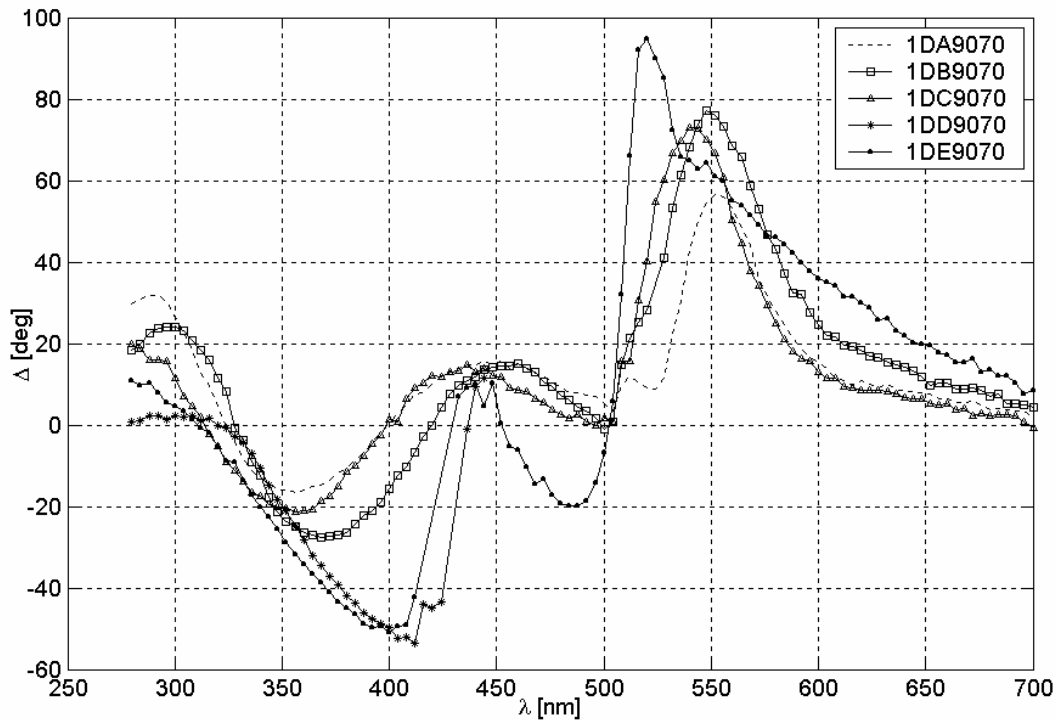


Fig. 3. Influence of groove breadth on dispersion of  $\psi$  ellipsometric angles

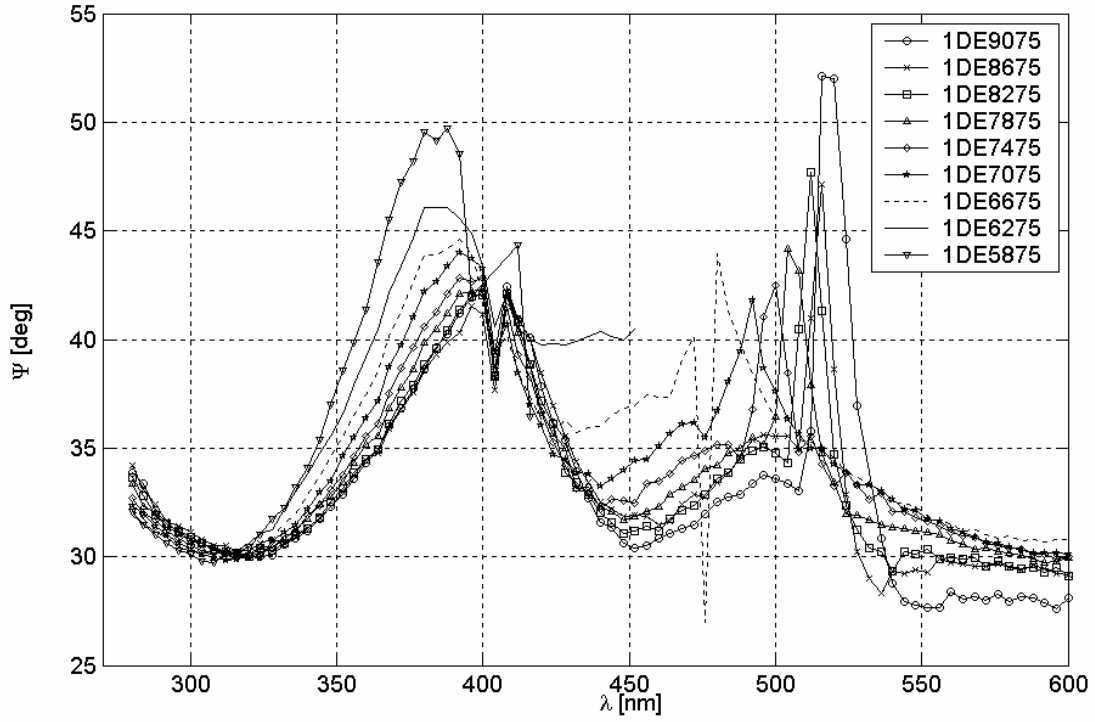
Figs. 3 and 4 demonstrate the distribution of spectral ellipsometric angles  $\psi$  and  $\Delta$  for the case when the incidence plane is normal to grating lamellas and groove breadths are different. The incident angle is fixed to be 70 degrees. As regard the  $\psi$  spectrum we can observe the sharp extremes located close to the wavelength of 525 nm (the depth of grooves). For the increasing of groove breadth the mentioned extremes (minima –

for A, B, C samples) are converted to sharp peak (E sample). It means that the ratio  $|r_p|/|r_s|$  abruptly increases. Because this change is connected with the level of fill factor (FF) the spectroellipsometry data can be used for the geometrical parameters specification of measured periodical structure. This effect brings the new possibilities in the realization of resonant polarization reflectors with defined narrow band reflectance and transmittance - together with multilayer twin systems. The dispersion curves of  $\Delta$  values related to inspect gratings intersect the zero  $\Delta$  level at some different wavelengths (Fig. 4). The concrete value of wavelength, which is characterized by the same phase for s- and p- polarized reflection coefficients, depends strongly on the fill factor of analyzed periodical structure. This tendency can be used in the shape testing of gratings by spectral ellipsometry. To compare  $\psi$  and  $\Delta$  angles of the collection of samples with different values of FF it can be observed that the sharp peaks positions for the grating with the lowest value of fill factor (E sample) are characterized for  $\psi$  and  $\Delta$  by 512 nm and 521 nm, respectively, in wavelength scale.

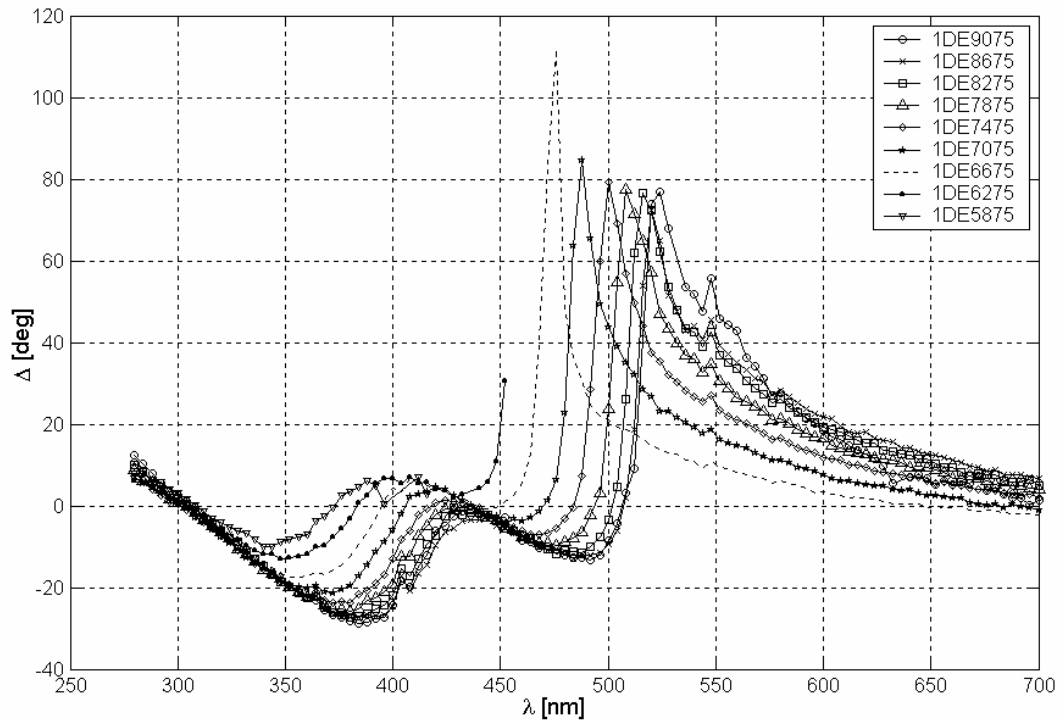


**Fig. 4.** Influence of groove breadth on dispersion of  $\Delta$  ellipsometric angles

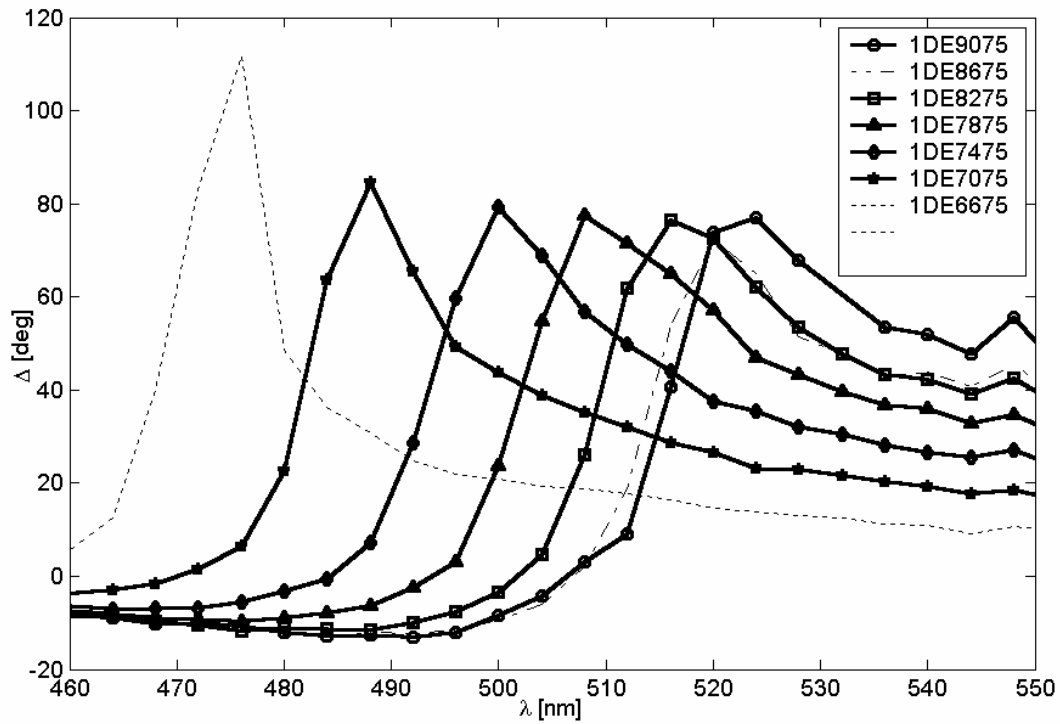
The effects of mutual position of incidence plane and lamellar edges are experimentally documented on the following figures. Fig. 5 describes  $\psi$  distribution as function of wavelength at 75 degrees incident angle for E sample. The symbolic description “90” means orthogonal mutual configuration of incidence plane and lamellas. For parameters characterizing the discussed sample (E) the experiments have been realized from 90 to 58 degrees. For lower angles (the plane of incidence is moved closely to lamellar edges) the spectral ellipsometry measurement has been displayed by some instability and serious results can be expected only for short wavelengths related to our spectral band. For normal mutual configuration incidence plane and lamellas the resonant peak is located to the wavelengths of 518 nm (near to the groove depth – 525 nm). If this mutual angle is decreasing this maximum of  $\psi$  dispersion is shifted to shorter wavelength. This tendency is confirmed by  $\Delta$  distribution (Fig. 6). The detail of this process is documented on Fig. 7. Here for starting mutual angle (90 degrees) the resonant maximum can be observed at 524 nm (the declared depth of grooves is 525 nm) and at 66 degrees relevant maximum occurs at wavelength of 476 nm. As result in average for one degree mutual rotation the peak shift of two nanometers in  $\lambda$  scale is generated. Because the wavelength dependences of  $\Delta$  angles can be specified by applied arrangement of spectral ellipsometry with experimental accuracy better than by one nanometer the tested grating is attractive element for the construction of rotation sensor. The application of lamellar gratings offers the possibility of speed and non-contact turning measurements.



**Fig. 5.**  $\psi$  distribution as function of mutual position of incidence plane and grating edges

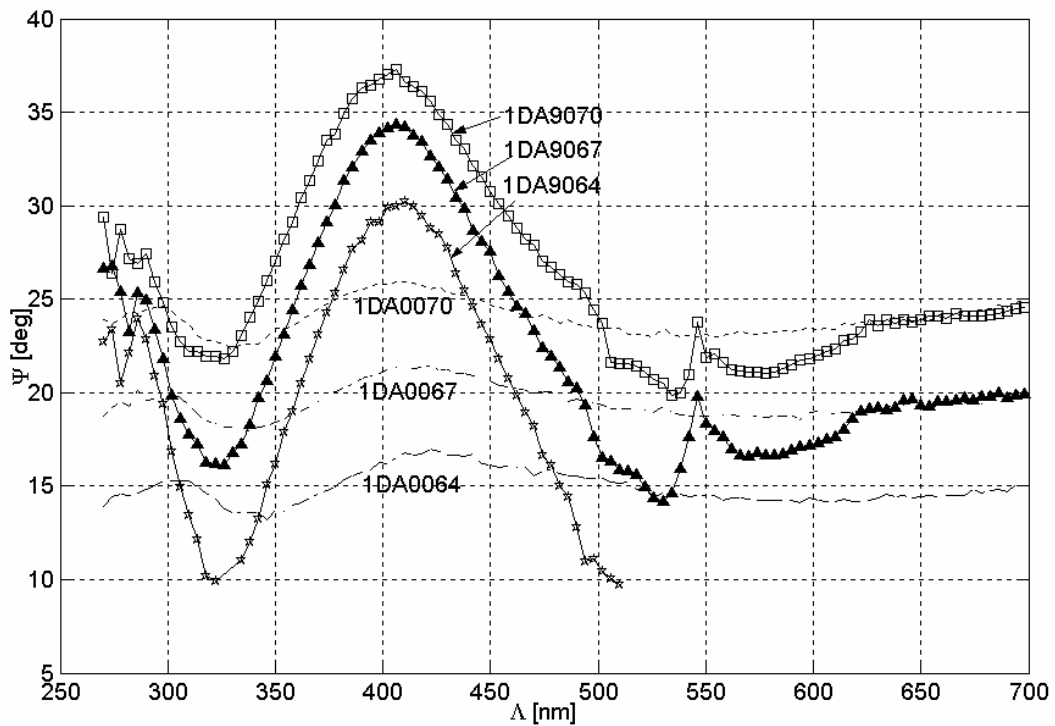


**Fig. 6.**  $\Delta$  distribution as function of mutual position of incidence plane and grating edges



**Fig. 7.**  $\Delta$  distribution as function of mutual position of incidence plane and grating edges - detail

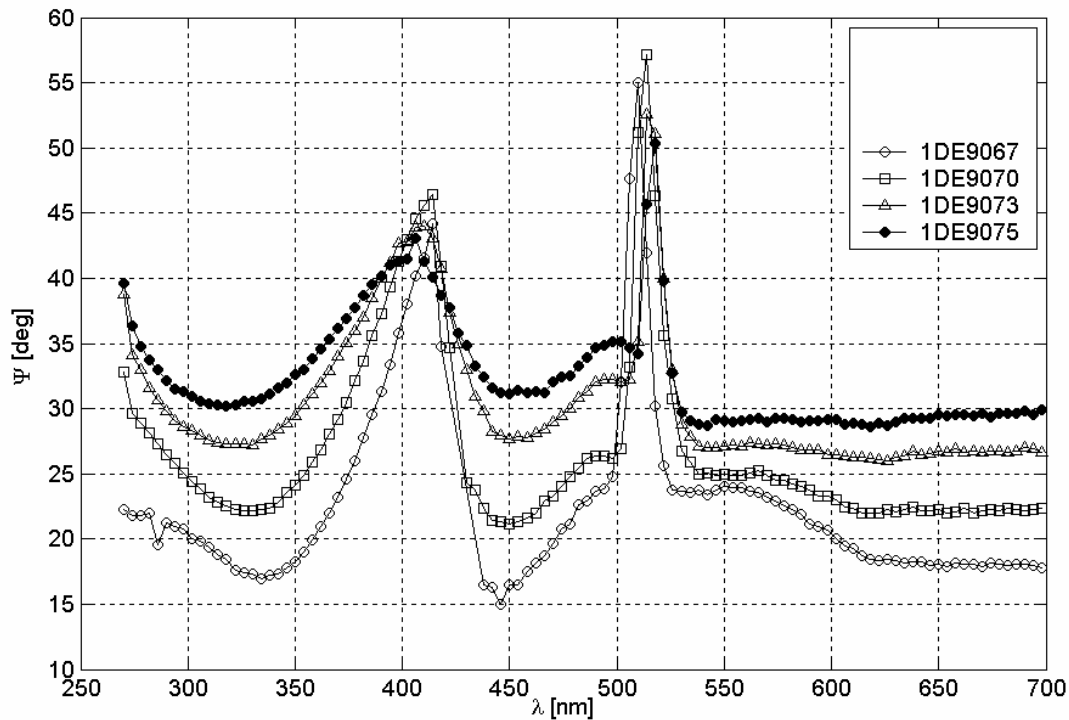
The influences of limit mutual angle position (normal versus parallel) are documented for A sample on Fig. 8. One half of curves for three different incidence angles (64, 67, 70 degrees) are related to orthogonal geometry (solid lines). The second one indicated by dashed lines describes  $\psi$  dispersion for parallel mutual configuration of incidence plane and lamellas. The amplitude modulation – in agreement with electromagnetic field theory – affects  $\psi$  angles only for orthogonal geometry. The parallel configuration



**Fig. 8.** Comparison of  $\psi$  dispersion for normal and parallel mutual geometry of incidence plane and lamellas

is characterized by insignificant changes of discussed ellipsometric data. For the longest wavelengths of our spectra the curves related to limit angle position of incidence plane and grating relief practically flow together for the same incidence angle of light beam.

The last figure (No. 9) documents the incidence angle influence on  $\psi$  angles for E sample. The sharp resonant peaks for incidence angles of 75, 73, 70, and 67 degrees are accumulated around wavelength 510 nm. The shifts of these maximum values for mentioned angles are within frame of some nanometers only. It is in contrast with dependences obtained for the situation when the angle motion (rotation) of incident plane has been analyzed (Figs. 5, 6, 7). It means that the groove depth of grating can be relatively easy specified by spectral ellipsometry. Of course we have to include the incidence angle corrections.



**Fig. 9.** Incidence angle influence on  $\psi$  angles for E sample

## Conclusions

The experimental study of deep lamellar gratings by spectral ellipsometry opened the progressive application possibilities of these structures and supported the theoretical approaches for the modelling of these periodical systems. The diffraction properties of the deep one-dimensional gratings are characterized by high sensitivity to breadths of grooves. The resonant character of this effect is typical for wavelengths close to relief depth. The important attribute is the influence of mutual position of incidence plane and grating geometry on polarization quality of reflected beams. This effect offers the promising applications in measuring technique. Of no little importance are the experimental evidences that the spectral ellipsometry is effective and accuracy inspection approach for the specification of geometrical parameters of above mentioned structures with 1D periodic ordering.

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## Resumé

Experimentální spektrální elipsometrie jednodimenzionálních hlubokých lamelárních mřížek

Fázově modulovaná spektrální elipsometrie (PMSE) jako vynikající experimentální technika pro určování optických a geometrických parametrů tenkých vrstev a dielektrických povrchů tvořených multivrstvami byla využita pro výzkum jednorozměrných hlubokých mřížek. Jako světelný zdroj byla použita Xe-Hg lampa. Kombinace dvojmřížkového monochromátoru a fotonásobiče tvoří nulovací detektor. Polarizační stavy dopadajících a odražených světelných svazků byly modulovány křemennými hranoly Rochonova typu. Otočný achromatický čtvrtvlnný retardér kompenzoval fázové posuvy odražené vlny. Experimenty byly prováděny pro úhly dopadu od 48 do 75 stupňů (v závislosti na druhu měření) pro nulový reflexní řád ve spektrální oblasti 270 až 700 nanometrů.

Lamelární mřížky byly připraveny leptací metodou na společné podložce z oxidu křemíku. Perioda a hloubka vrypů byly u všech vzorků jednorozměrných mřížek stejné a činily 260 nm respektive 525 nm. Vzorky se odlišují šířkou vrypů. Hlavní pozornost byla soustředěna na experimentální studium vlivu šířky vrypu, vzájemné polohy roviny dopadu světla a mřížkové geometrie a úhlů dopadu světla na disperzi elipsometrických úhlů difraktovaného světla.

V případě, že dopadající vlna je kolmá k lamelám periodické struktury, pozorujeme výrazné extrémní disperzní křivky  $\psi$  v okolí vlnové délky 525 nm. S rostoucí šířkou vrypů původně minima průběhů elipsometrického úhlu  $\psi$  přecházejí ve výrazné maximum. Disperzní závislosti rozdílů fází reflexních koeficientů pro p a s polarizované vlny ( $\Delta$  elipsometrické úhly) vykazují jednak v daném vlnovém spektru několikrát soufázovost, jednak lze pozorovat posuv extrémů  $\Delta$  v závislosti na šířce mřížkových drážek.

V případě, že při konstantním úhlu dopadu měníme vzájemnou polohu roviny dopadu a lamel mřížek, lze pozorovat posuvy poloh extrémů jak pro  $\psi$  disperzi, tak také pro průběhy  $\Delta$ . Tato měření, vzhledem k tomu, že mřížky mají charakter „hlubokých“ struktur, byla prováděna v úhlovém intervalu 90 (kolmost roviny dopadu a lamel) až 58 stupňů. U  $\Delta$  parametru průměrná hodnota posuvu maxima činí 2 nanometry ve vlnovém rozsahu při otočení roviny dopadu o 1 stupeň. Tento fakt otevírá aplikační možnost uvedených struktur jako inkrementálních optických čidel.



Zajímavou se také ukazuje závislost vlnové disperze  $\psi$  na úhlu dopadu. Příslušné křivky jsou charakterizovány ostrými extrémy v okolí 525 nm (deklarovaná hloubka vrypů) pro daný interval úhlů dopadu světla.

Ze získaných experimentálních dat vyplývá, že pomocí spektrální elipsometrie lze na jedné straně určovat geometrické rozměry hlubokých mřížek, na straně druhé se zde nabízí možnost pro optimalizaci progresivních optických senzorů polohy a rotace.

Recenzent: Doc. RNDr. Petr Hlubina, CSc., Ústav fyziky FPE SU Opava

