

Planar Quarter Wave Stack Reflectors Prepared from Chalcogenide Ge-Se and Polymer Polystyrene Thin Films

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1. Introduction

For many decades the multilayer structures consisting of alternating dielectric films with sufficient differences in the refractive index have played an important role in designing highly effective planar optical elements, namely mirrors and filters. The one-dimensional photonic bandgaps are formed in the dielectric multilayers if optical constants and thicknesses of films meet the Bragg resonance condition. These photonic bandgaps display themselves as high intensity reflection or transmission bands in the optical spectra of the dielectric multilayers. Dielectric multilayers are widely used as highly effective reflectors and filters in current optical devices. Their bandgap structure can be predicted according to the theory of the light propagation through stratified dielectric media (Yeh et al., 1977) and their optical properties then adjusted appropriately.

The interest in dielectric multilayers was renewed again when the omnidirectional bandgap (Fink et al., 1998) was reported for the reflectors having the refractive index difference of films higher than $\Delta n \geq 0.8$. The omnidirectional total reflection from such quarter wave stacks (QWS) allows the designing of the reflectors that can reflect the light at any angles and polarizations. A common goal in current QWS device design is then a maximization of a bandwidth of the omnidirectional bandgap by using materials having a high refractive index difference.

From material point of view, the first successful fabrications of planar omnidirectional reflectors with bandgaps in the visible, near-infrared and middle-infrared ranges were reported on materials such as $\text{SiO}_2/\text{TiO}_2$, Si/SiO_2 or GaAlN/GaN . These materials exhibited favourable optical properties and a high chemical, thermal, and mechanical stability. However, they brought at the same time high cost and some technological difficulties relating to their high temperature processing (annealing) which can lead to the structural defects of films, e.g., crystallization, cracking or delamination (Chen et al., 1999 and Deopura et al., 2001).

Recent development also showed a possibility of a low temperature fabrication of QWS devices from chalcogenide and polymer films. Chalcogenide glasses and organic polymers

seem to be a good alternative to the above-mentioned dielectric materials and deposition methods because they require processing at temperatures below $T < 200^\circ\text{C}$, offer a sufficiently high refractive index difference and also exhibit favourable mechanical properties, see already published results (Fink et al., 1998; Decorby et al., 2005; Clement et al., 2006; Kohoutek T. et al., 2007; Abouraddy et al., 2007 and Kohoutek T. et al., 2008^a). As shown in the literature (Decorby et al., 2005 and Abouraddy et al., 2007), the omnidirectional QWS devices can be fabricated as planar or scrolled multilayers using thermal evaporation (TE) of high index As-Ge-Se or As-Se chalcogenide glasses and the spin-coating (SC) of PAI or PES polymers which form a low index films. A combination of highly transparent chalcogenide and polymer films enables these QWS elements to cover the wavelength ranges reaching from the visible to the infrared spectral regions including telecommunication (1.3-1.7 μm), security laser ($\sim 2.8 \mu\text{m}$), night vision (3-5 μm) or even high-power laser (~ 5.4 and $\sim 10.6 \mu\text{m}$) wavelengths.

In this paper we present the preparation and determination of the optical properties of dielectric reflectors which consisted of eight chalcogenide $\text{Ge}_{25}\text{Se}_{75}$ films periodically alternating with seven polymer Polystyrene (PS) films. The reflectors were designed with the first order normal incidence bandgap at a telecommunication wavelength of $\lambda = 1.55 \mu\text{m}$ and the refractive index difference between the used films was ~ 0.821 . We also studied the optical properties of the Ge-Se/PS reflector deposited on $\sim 40 \text{ nm}$ gold film to show the effect of metal layer on the stopband of a newly formed metal/dielectric reflector.

Keywords

Polymers, Chalcogenide glasses, Dielectric multilayers, Optical properties

2. Experimental

Deposition of Reflectors

The Ge-Se/PS reflectors were fabricated as one-dimensional photonic crystals from alternating high index chalcogenide (eight layers) and low index polymer (seven layers) films as schematically illustrated in Figure 1. The film thickness values necessary to centre the first order normal incidence bandgap of the reflectors near the telecommunication C-band ($\lambda \sim 1.55 \mu\text{m}$) were calculated according to the QWS condition, $d_i = \lambda/4 n_i$ (Kim S.-H. & Hwangbo C.K., 1998), knowing the appropriate values of film refractive index of individual Ge-Se and PS films.

Polystyrene films with refractive index $n_{\lambda = 1.55 \mu\text{m}} \sim 1.531$ were prepared toluene solutions where 0.72 g of polymer pellets (Sigma-Aldrich, $M_n = 130\text{k}$, $M_w = 240\text{k}$) were dissolved in 10 ml of pure toluene using a spin-coating technique. The demanded thickness of polystyrene films ($d \sim 250 \text{ nm}$) was achieved by spinning a drop of the solution for 50 seconds at a spin speed of 5000 rpm. We carried out the calibration of film thicknesses as a function of solution concentration and spin speed before multilayer preparation. After deposition of each polymer PS film, the films and then the multilayers were thermally stabilized in a vacuum oven at $T = 90^\circ\text{C}$ and $p \sim 5 \text{ Pa}$ for one hour to remove solvent residua.

Before the deposition of chalcogenide films, the bulk glass with a composition of $\text{Ge}_{25}\text{Se}_{75}$ was prepared by a standard synthesis from high purity (99.999 at. %) elements in sealed evacuated quartz ampoule placed in a rocking furnace ($T = 990^\circ\text{C}$, 24 hours) followed by air quenching of the melt. Pieces of bulk glass were placed in a corundum crucible and heated

in a vacuum chamber. Chalcogenide Ge-Se films with a thickness of $d \sim 165$ nm and refractive indices $n_{\lambda = 1.55 \mu\text{m}} \sim 2.352$ were then thermally evaporated onto chemically pre-cleaned silica glass substrates (1×1 inch). The substrates were fixed to a planetary rotation system which improved the homogeneity of film thicknesses during evaporation. The deposition rate was ~ 5 nm/s and residual pressure in the vacuum chamber $p \sim 10^{-4}$ Pa. The film thicknesses and evaporation rates were measured by a calibrated quartz crystal monitor.

Some of the multilayers were fabricated on substrates coated in advance with a thermally evaporated gold layer with a thickness of $d \sim 40$ nm to study the influence of metal on the optical properties of the newly formed metal/dielectric reflector.

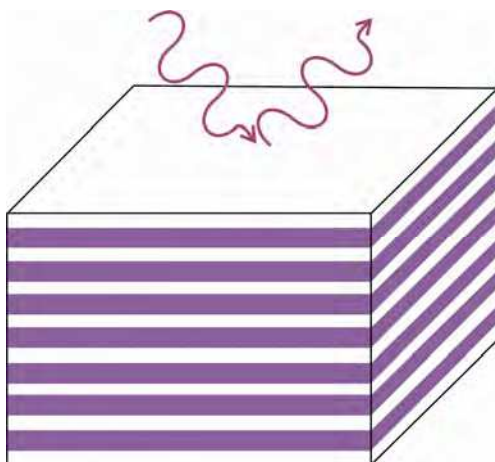


Fig. 1. A schematic sketch of the intended chalcogenide/polymer reflector.

Measurement of Optical Properties

The optical properties of the fabricated reflectors were investigated by means of an optical spectroscopy. The normal incidence reflectivity was recorded by a Jasco V-570 UV-vis-NIR spectrophotometer in the spectral range of 400-2300 nm with a resolution of 2 nm. The experimental ellipsometric data of individual Ge-Se and PS films were recorded using a spectral ellipsometer VASE, J. A. Woollam Co. Inc. with NIR at angles of incidences 60, 65 and 70 ° and in the spectral range of 300-2300 nm. The optical functions $n(\lambda)$ and thicknesses of the studied chalcogenide and polymer films were evaluated by fitting the ellipsometric Ψ and Δ spectra using the Tauc-Lorentz dispersion formula in the entire spectral region (according to Jellison G.E & Kosine F.A., 1996). The Cauchy formula was used to fit the optical functions of the studied samples in the transparent region (Tompkins H.G & McGahan W.A., 1999). Knowing the optical parameters and thicknesses of films, we were able to generate the optical reflectivity spectra of the intended dielectric reflectors which consisted of eight Ge-Se + seven PS layers for both the normal and the oblique light incidence and for TE- and TM-polarizations, respectively.

3. Results

Dielectric and Metal/Dielectric Reflectors

The fabrication of Ge-Se/PS reflectors was followed by studying their cross-section to confirm if the film order and thickness accuracy in the multilayer were correct. The cross-section image of the multilayer was captured using the JEOL JSM-5500LV scanning electron microscope SEM after a rupture of the prepared reflector across a groove made by a diamond tip. The periodically alternating films made from chalcogenide glass (brighter stripes) and polystyrene (wider and darker stripes) can be then seen in Figure 2. Chalcogenide films appeared brighter in the image because of their low electron conductivity. The lower conductivity causes the films to be locally charged by the electrons gathered in the scanned area. The polymer films have slightly higher electron conductivity than amorphous chalcogenide films and therefore appear darker in the SEM image. Although the film order in the reflector is clearly apparent from the image, the resolution of our microscope did not allow us to focus on the details of film boundaries. To achieve a higher image resolution, it is necessary to observe the samples cross-sectionally using a high resolution transmission electron microscopy (HR-TEM) (Kohoutek et al.^b, 2008 and Kohoutek et al., 2009).

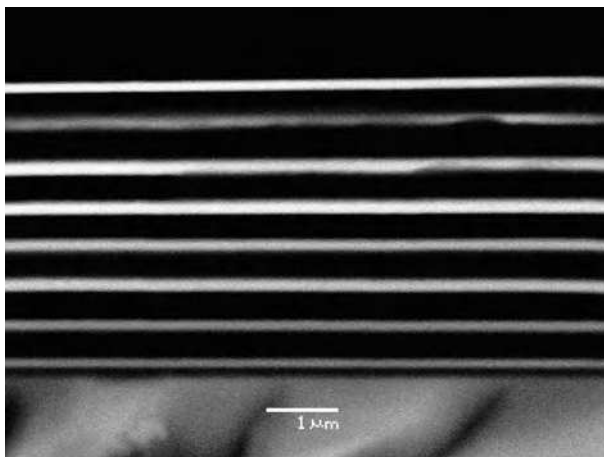


Fig. 2. The SEM image shows a cross-section of Ge-Se/PS reflector. The darker and thinner films belong to polystyrene while brighter and thicker layers to Ge-Se films.

The optical properties of chalcogenide Ge-Se and polymer PS films were studied using spectral ellipsometry. This method is very useful for the determination of the optical parameters of thin films and their thicknesses. Figure 3. shows the optical function $n(\lambda)$ of Ge-Se and PS films in the spectral range of 500-1700 nm. The difference in the index of refraction between Ge-Se ($n = 2.352$) and PS ($n = 1.531$) films was ~ 0.821 at $\lambda = 1.55 \mu\text{m}$. This wavelength was intended for the stopband of the prepared reflectors. As the absorption of films is expected to be in the visible part of the electromagnetic wavelengths it is very unlikely that it plays a major role to the reflector stopband in the near infrared range of $\sim 1.55 \mu\text{m}$. As mentioned above, the values of film thicknesses, $d_{\text{Ge-Se}} = 165 \text{ nm}$ and $d_{\text{PS}} = 250$

nm, were calculated according to the QWS condition before the reflector deposition. The thickness of each prepared individual film was then checked using spectral ellipsometry. The average inaccuracy in film thickness was found to be ± 7 nm (~ 4.2 %) for chalcogenide and ± 17 nm (~ 6.8 %) for polymer films. A similar inaccuracy in the film thicknesses was also reported in the literature (DeCorby et al., 2005).

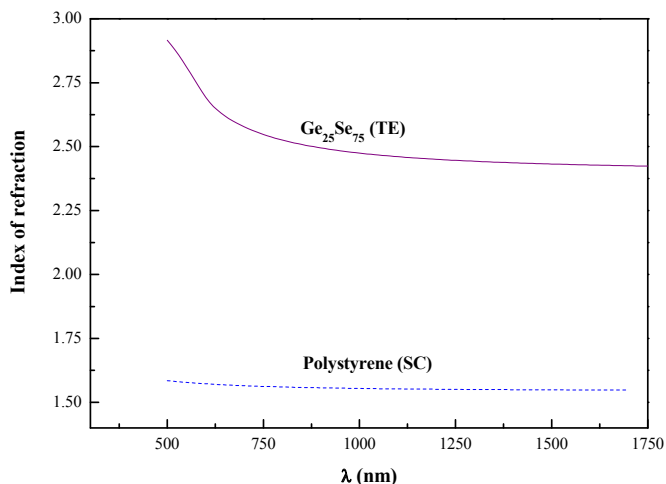


Fig. 3. The films refractive indices, i.e., $n(\text{Ge-Se})_{\lambda = 1.55 \mu\text{m}} \sim 2.352$ and $n(\text{PS})_{\lambda = 1.55 \mu\text{m}} \sim 1.531$, were determined from optical functions $n(\lambda)$ of the films according to the ellipsometric measurements.

The normal incidence optical reflectivity of Ge-Se/PS and Au/Ge-Se/PS reflectors was studied using an UV/vis/NIR spectroscopy in the spectral range of 400-2300 nm. Figure 4. represents the comparison between the measured optical reflectivity of the dielectric reflector (a) and the metal/dielectric reflector (b) and theoretical optical reflectivity of both reflectors obtained by modelling ellipsometric parameters of a complex system of 15 or 16 layers and the substrate. The normal incidence stopband with the optical reflectivity higher than $r \geq 98.5$ % was found to be in the range of 1527-1730 nm with a maximum at $\lambda = 1588$ nm, $r = 99.09$ %. In the same spectra is seen both near infrared first order stopbands of Ge-Se/PS reflector and also the second order stopbands near $\lambda = 800$ nm. The formation of the second order stopbands reflects a high order and a good accuracy of the fabricated reflectors. By adding a ~ 40 nm thick gold layer between the reflector and the substrate, the stopband splits into two parts separated by a ~ 30.55 % transmission band with a maximum at $\lambda = 1772$ nm. The stopband splitting was also accompanied by an increase in the maximum optical reflectivity of the metal/dielectric reflector up to $r \geq 99.41$ % at $\lambda = 1553$ nm and the broadening of the stopband bandwidth in the range of 1464-1685 nm, where $r \geq 98.5$ %. The effect of gold layer on the reflector stopband is apparent in Figure 5., where details of the above-mentioned increase in optical reflectivity and the band splitting of the first order stopbands of dielectric and metal/dielectric reflectors are shown.

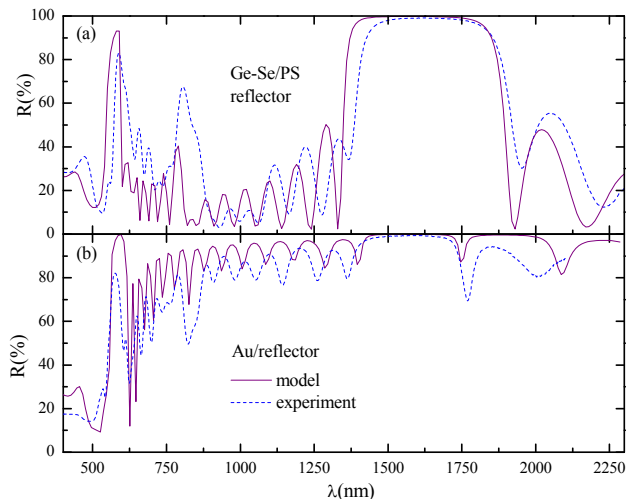


Fig. 4. The comparison of the experimental and modelled normal incidence optical reflectivity is shown for Ge-Se/PS reflector (a) and Au/Ge-Se/PS reflector (b).

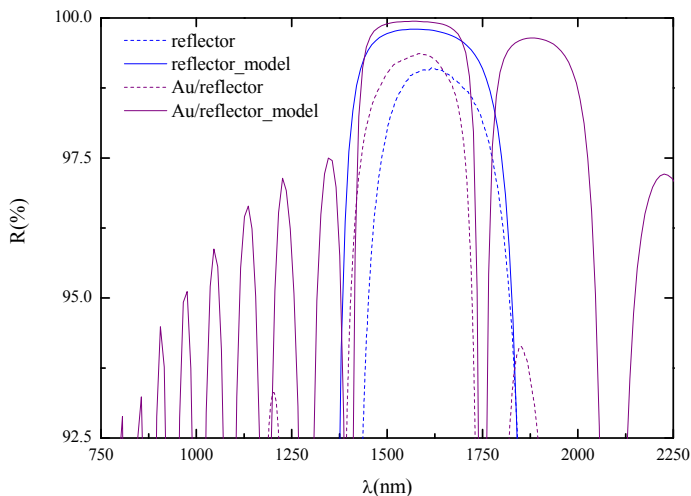


Fig. 5. The optical reflectivity spectra of Ge-Se/PS reflector and Au/Ge-Se/PS reflector show reflectivity maxima of normal incidence stopbands in detail, i.e., an increase in the optical reflectivity of metal/dielectric reflector compared to the maximum achieved for Ge-Se/PS reflector.

Based on our ellipsometric study of the parameters of individual chalcogenide and polymer films, we can demonstrate the behaviour of Ge-Se/PS and Au/Ge-Se/PS reflectors at oblique angles of incidence. The optical reflectivity spectra of the reflectors were modelled for oblique angles of 35° , 45° and 55° and transverse magnetic (TM) and transverse electric (TE) modes of the light. As apparent from Figure 6., the stopband position of the Ge-Se/PS reflector shifted towards lower wavelengths (a blue-shift) with an increasing angle of propagating light. The shift was more apparent in the reflectivity of the light in TM-mode, especially in its longer wavelength spectra, as TM-mode defines the low frequency edge of the stopband. Such a shift is also in an agreement with theoretical predictions (Yeh et al., 1977). The first order bandgap of the TM mode decreases with an increasing angle of the light incidence, i.e., $\Delta\lambda = 265, 196$ and 76 nm for $r \geq 98.5\%$. The bandgap of the TE mode increases at the same time, i.e., $\Delta\lambda = 417, 442$ and 468 nm. A similar angular dependence of the optical properties observed for the first order bandgap of the dielectric reflector can also be seen for the second order bandgaps near 750 nm. In comparison with Ge-Se/PS dielectric reflectors, modelled angular optical reflectivity spectra of metal-dielectric Au/Ge-Se/PS reflectors revealed a partially different behaviour, see Figure 7. An effect of the blue-shift of the bandgap and its broadening at higher oblique angles is similar. However, an addition of a gold layer to the dielectric reflector forms a new transmission band in the middle of the original bandgap which becomes highly intensive in the TM-mode as the angle of the light incidence increases, i.e., $t = 28.19\%$ for $\lambda = 1683$ nm and 35° , $t = 42.28\%$ for $\lambda = 1640$ nm and 45° and $t = 64.65\%$ for $\lambda = 1595$ nm and 55° , respectively. In the TE-mode, the transmission band subsequently vanishes at higher oblique angles, i.e., $t = 7.64\%$ at $\lambda = 1691$ nm and 35° , $t = 4.57\%$ at $\lambda = 1653$ nm and 45° and $t = 2.65\%$ at $\lambda = 1613$ nm and 55° , respectively.

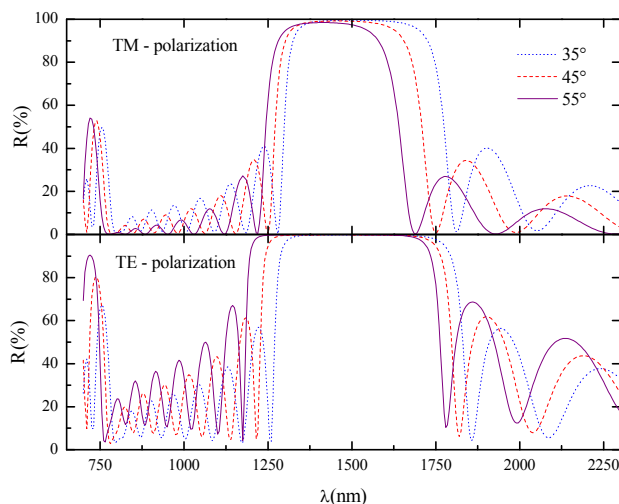


Fig. 6. Comparison of the modelled optical reflectivity of Ge-Se/PS reflector is shown for the light with TM- and TE-polarization at oblique angles 35° , 45° and 55° .

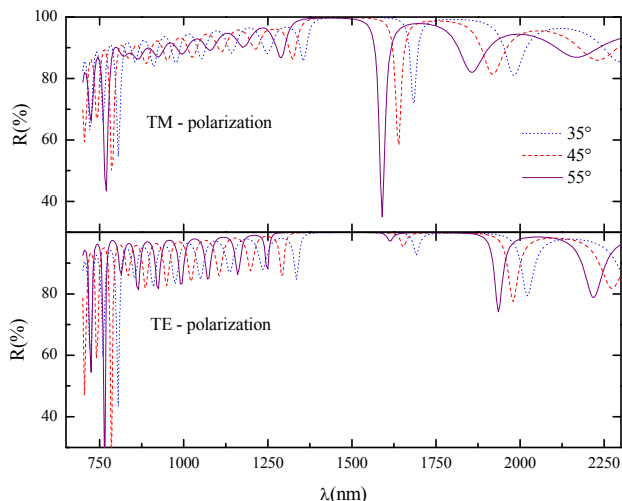


Fig. 7. The modelled optical reflectivity of Au/Ge-Se/PS reflector documents a broadening, blue-shift and stopband splitting of the bandgap of metal/dielectric reflector at oblique angles of 35°, 45° and 55° and TM- and TE-polarized light.

4. Discussion

Dielectric and Metal/dielectric Reflectors

In this study, we have shown that dielectric and metal/dielectric reflectors with the optical reflectivity higher than $r > 99\%$ can be fabricated using low-temperature and technologically simple deposition techniques from chalcogenide glasses and polymer films. We aimed rather at an investigation of the optical properties of the used materials and dielectric and metal/dielectric reflectors, than at the fabrication of reflectors with the highest practically achievable optical reflectivity, which would of course demand a much higher number of deposited layers.

The factors determining the quality of Ge-Se/PS reflectors and dielectric reflectors in general are an inaccuracy in thin film deposition, homogeneity of film optical properties and their surface roughness. The inaccuracy in a film deposition was ± 7 nm for chalcogenide and ± 17 nm for polymer films according to ellipsometric results. It represents roughly a $\sim 5\%$ deviation in thickness. In our opinion, this value can be further reduced maximally up to the level of 1-2% especially if we consider that the main issue remains the thickness homogeneity of spin-coated films across their entire surface. For comparison, the inaccuracy in thickness for crystalline films prepared using more sophisticated and more expensive vacuum deposition methods such as an e-beam or CVD is under 1% (Yao et al., 2007 and Perez et al., 2005). In case of 1D-photonic crystals, i.e., multilayers a film thickness inaccuracy in order of percent units has basically a low impact on the optical properties of such multilayers.

On the other hand, the optical properties of films and multilayers can be strongly influenced by their eventual compositional inhomogeneity across the entire film thickness. This factor is very important especially for thermally evaporated chalcogenide films. It is well known that an effect of phase separation often occurs in chalcogenide glasses and their thin films including Ge-Se system (see Borisova Z., 1981 and Orava J. et al., 2009). Regarding the fabricated reflectors, a phase separation in Ge-Se films was minimized using a relatively high evaporation rate ~ 5 nm/s during deposition. The higher deposition rate means higher energy brought on the bulk glass placed in a crucible and its quicker evaporation which prevents a phase separation. In our opinion, a compositional inaccuracy of amorphous films in order of several tenths of per cent is acceptable if we consider that properties, e.g., optical or electrical, of glassy and amorphous (disordered structure) materials change very slightly with such a change in composition. It is in sharp contrast to crystalline (ordered structure) materials where any slightest compositional change causes a considerable change in material properties. Freshly prepared (as-deposited) chalcogenide films also underwent an annealing procedure at $T = 90$ °C which is necessary for their stabilization. This temperature is, in fact, not appropriate (T_C of Ge-Se film is much higher) but limited by thermal stability of PS films in the reflector.

The root mean square surface roughness of individual Ge-Se and PS films, R_q , determined from atomic force microscopy (AFM) data was in the range of $R_q \sim 2-5$ nm. These values were lower than the surface roughness of oxide sol-gel films $R_q > 10$ nm (reported in Almeida & Rodrigues, 2003) and slightly higher than values achieved by well defined e-beam deposition $R_q \sim 1$ nm (Yao et al., 2007). Higher surface roughness of films in photonic devices can cause undesired scattering of the light and noises which reduce their efficiency. An important issue for the multilayers is the quality of film boundaries, especially, if the multilayer structure is made from materials with different mechanical properties. Good adhesion, chemical stability and thermal expansion coefficient of films are also required.

The optical reflectivity of the fabricated dielectric reflector and metal-dielectric reflector was compared with modelled optical reflectivity obtained from ellipsometric results (see Orava et al., 2008). A strong correlation between these values is apparent in Figs. 4. and 5. Modelled curves predicted higher optical reflectivity of the metal-dielectric reflector compared to the Ge-Se/PS reflector near $\lambda = 1550$ nm and also the behaviour of both reflectors at oblique angles. An increase in the optical reflectivity of metal-dielectric reflector was then confirmed by the reflectivity measurement performed on the fabricated reflectors. In general terms an addition of metal layer between the dielectric reflector and the substrate leads to an increase in the optical reflectivity, blue-shift and broadening of the stopband. In our case, an addition of a ~ 40 nm gold layer to the dielectric multilayer caused a 0.32 % increase in the optical reflectivity of reflectors ($r = 99.09 \rightarrow 99.41$ %) and blue-shift of the stopband maximum ($\lambda = 1588 \rightarrow 1553$ nm) and bandwidth broadening $r \geq 98.5$ % ($\Delta\lambda = 203 \rightarrow 221$ nm). We should notice that a 0.32 % increase in the stopband maximum of the reflector represents really significant value if we consider the reflectivity range $r > 99$ %. From the application point of view, an addition of one thin metal layer can substitute a deposition of several pairs of dielectric films necessary to achieve the same value of optical reflectivity. On the other hand, the optical reflectivity of the multilayer increases significantly in the entire wavelength range, which reduces the difference between reflectivity maxima and minima. The metal/dielectric reflectors are then suitable namely for the applications where the highest possible reflectivity at the lowest number of deposited films is required.

Future prospects of Ge-Se/PS multilayers could be in their deposition on flexible polymer substrates (as reported by DeCorby et al., 2007) or in designing more complex optical devices, including Bragg reflectors (see the review by Abouraddy et al., 2007). Exploiting high transparency of chalcogenide and polymer films, the optical properties of the described dielectric multilayers can be tuned from the visible up to $\lambda = 3000$ nm (above this wavelength, the absorption of the stretching vibrations of C-C, C-H bonds in polymers can be found). The near infrared ($\lambda = 800$ -2500 nm) dielectric reflectors and filters can be then designed for applications in telecommunication (signal processing), moisture or dust detection in the air (astronomy, meteorology), measurement of oxygen in hemoglobin (oximetry, medicine), soft-laser frequencies (security lasers), etc.

5. Conclusions

In this study, we fabricated Ge-Se/PS dielectric and Au/Ge-Se/PS metal/dielectric reflectors from amorphous chalcogenide and polymer films with the optical reflectivity higher than $r > 99$ % near $\lambda \sim 1550$ nm using low-temperature and inexpensive deposition techniques. Our attention was paid to the determination of the optical properties of films and multilayers to demonstrate the suitability of the selected materials for designing 1D-photonic crystals. The main conclusion of the study is that highly intensive one dimensional photonic bandgaps easily tunable at any wavelength range in the near infrared ($\lambda \sim 800$ -2500 nm) can be achieved by using chalcogenide/polymer multilayers. It is mainly because of a sufficient difference in the refractive index of these materials and their high infrared transparency. Another important conclusion is that an addition of a metal layer to a dielectric reflector can further enhance the stopband maxima or reduce a number of necessary layer pairs in the reflector to reach the same optical reflectivity values. The QWS devices based on high refractive index chalcogenides and low refractive index polymers seem to be promising for applications demanding favorable performance ratio.

Acknowledgements

The authors thank the Research Centre (University of Pardubice, Project No. LC 523) and the Czech Republic Grant Agency (GA CR, Grant No. 203/08/P204) for their financial support of this study.

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Polymer Thin Films

Edited by Abbass A Hashim

ISBN 978-953-307-059-9

Hard cover, 324 pages

Publisher InTech

Published online 01, April, 2010

Published in print edition April, 2010

This book provides a timely overview of a current state of knowledge of the use of polymer thin film for important technological applications. Polymer thin film book covers the scientific principles and technologies that are necessary to implement the use of polymer electronic device. A wide-ranging and definitive coverage of this emerging field is provided for both academic and practicing scientists. The book is intended to enable readers with a specific background, e.g. polymer nanotechnology, to become acquainted with other specialist aspects of this multidisciplinary field. Part A of the book covers the fundamental of the key aspect related to the development and improvement of polymer thin film technology and part B covers more advanced aspects of the technology are dealt with nano-polymer layer which provide an up-to-date survey of current research directions in the area of polymer thin film and its application skills.

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Tomas Kohoutek, Jiri Orava, Martin Hrdlicka, Jan Prikryl, Tomas Wagner and Miloslav Frumar (2010). Planar Quarter Wave Stack Reflectors Prepared from Chalcogenide Ge-Se and Polymer Polystyrene Thin Films, Polymer Thin Films, Abbass A Hashim (Ed.), ISBN: 978-953-307-059-9, InTech, Available from: <http://www.intechopen.com/books/polymer-thin-films/planar-quarter-wave-stack-reflectors-prepared-from-chalcogenide-ge-se-and-polymer-polystyrene-thin-f>

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