Determination of stress-optical and thermal-optical coefficients of Nb$_2$O$_5$ thin film material

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The stress-optical and thermal-optical coefficients of Nb$_2$O$_5$ in thin film narrow bandpass filters (NBPFs) are determined by using the experimental data of temperature-dependent shift of center wavelength. The five-cavity subnanometer NBPFs under consideration are made by the same Nb$_2$O$_5$ and SiO$_2$ film materials but deposited on two different substrates (WMS-02 and F-7) through ion-assisted deposition process. The shift of center wavelength in the bandpass filters due to temperature rise is theoretically formulated and related to a variety of factors including the change of refractive index and film thickness due to temperature rise as well as thermal stress. By using the experimental data of center wavelength shift, the unknown stress-optical and thermal-optical coefficients of Nb$_2$O$_5$ thin film material are evaluated. These coefficients are equal to $-95.1 \times 10^{-12}$ Pa$^{-1}$ and $1.43 \times 10^{-5}$ °C$^{-1}$, respectively, at 1550 nm wavelength. © 2007 American Institute of Physics. [DOI: 10.1063/1.2435796]

I. INTRODUCTION

Thin film materials have important applications in diverse fields, such as microelectronics, optoelectronics, micromechanics, and optical sensors. In the field of communications, the technology of optical thin film deposition can now provide dense wavelength division multiplexing (DWDM) devices with excellent performance, reliability, and thermal stability, as well as low loss. Recently, thin film filter technology has gradually come to dominate the 200 and 100 GHz DWDM markets. Competing solutions, such as arrayed waveguide grating (AWG) and fiber Bragg grating (FBG) technologies, have trailed behind as a result of system complexity and manufacturing difficulties. Very good performance of thin film DWDM filters absolutely relies on the coating uniformity, residual stress, and physical properties of thin film materials, especially, as refractive index, Young’s modulus, Poisson’s ratio, and stress-optical and thermal-optical coefficients. Consequently, the accurate evaluation of the film properties is necessary in designing and fabricating complex devices.

The physical properties of a thin film material are closely correlated to its density. For instance, with a higher energy bombardment in the film deposition process, the associated densification process will yield the higher values for the refractive index, especially desired for optical applications. Thin dielectric films are generally deposited by a variety of deposition techniques, especially by ion-assisted deposition (IAD) and ion beam sputtering deposition (IBSD) processes, which involve a transport phenomenon of high energy materials in a relatively cold and low-pressure environment and will result in an amorphous film with high packing density, low optical scattering, and smooth surface with good mechanical and optical qualities. When the optical thin films cool down to room temperature from deposition temperature or expose to environments during service, significant stresses, deformations, and interface delamination may be introduced in the structure due to porosity, defects, intrinsic stress, and different thermal expansion coefficients between layers. Consequently, the quality and the optical performance of the devices may thus be sincerely deteriorated or damaged due to, for instance, the temperature and stress induced shift of center wavelength or degradation of transmission curve in the optical spectrum.

The total residual stress induced in a thin film material is generally composed of intrinsic stress and thermal stress. The former is generated during film growth due to relatively complicated microscopic mechanisms, such as the different spacings of atoms in a growing film, the incorporation of excess vacancies, the presence of impurities and bombardment by energetic particles, etc., while the latter results from the difference in the thermal expansion coefficients between adjacent layers. Residual stresses induced in the thin film material significantly influence not only the mechanical performance of coatings such as spallation resistance, thermal cycling life, and fatigue properties but also the optical, electrical, and magnetic behaviors of layer devices due to the...
cracking, interfacial delamination, and the change of physical properties due to their stress and deformation dependence in nature.

Recently, niobium (Nb) has become the focus of attention as a candidate alternative material to tantalum (Ta), both are in the same group of elements in the Periodic Table. For instance, Nb₂O₅ is a more promising material for electrolytic capacitors than Ta₂O₅. Moreover, Nb₂O₅ is an excellent candidate for optical thin film applications. It is transparent and has a high index of refraction in the wavelength range from 380 nm to 9 μm. In addition, it is insoluble in water, stable in air, relatively unreactive plus acid, and base resistant. However, very limited data on Nb₂O₅ thin films have been reported, especially the properties relevant to the stress-optical and thermal-optical coefficients.

Temperature stability of the center wavelength in thin film narrow bandpass filters (NBPFs) is always an important issue and has been investigated by several groups in recent decades. For instance, Takashashi developed an elastic strain model to investigate the temperature stability of four types of single-cavity, thin film NBPFs produced by IAD. The results revealed that the main reason why the temperature stability of the center wavelengths exhibits substrate dependency is due to a reduction in film packing density brought about by volumetric distortion of the film, which is caused by stress induced from the substrate.

The main objective of this work is to propose and provide a realistic analytical method to evaluate the stress-optical and thermal-optical coefficients of Nb₂O₅ thin film material by individually measuring the shift of center wavelength in two different NBPFs, made of the same film materials but deposited on two different substrates, respectively. These two kinds of substrates, i.e., WMS-02 and F7, have very similar coefficients of thermal expansion but a slight difference in Young’s modulus; however, two completely different shifts of central wavelength, one is the blueshift and the other is the redshift, are observed in the experiment as the temperature increases. By using the analytical model, the stress-optical and thermal-optical coefficients of Nb₂O₅ thin film material are accurately evaluated. Moreover, the detailed mechanisms in the central wavelength shift of NBPF are investigated and clarified.

II. EXPERIMENT

A. Filter design

The bandwidth requirements for a 100 GHz filter are 0.4 nm at −0.5 dB and 1.2 nm at −25 dB. The figure of merit, which is the ratio of the bandwidth at −25 dB to that at −0.5 dB, is 3.0. To manufacture a high-performance NBPF, a five-cavity Fabry-Pérot-type filter with 187 layers coated on the top side of the substrate is necessary. A four-layer anti-reflective coating is coated on the back side of the substrate to prevent étalon-effect–induced transmission ripples from affecting the measurements. Thus the sequence of the top-side multilayer is designed by the following scheme:

\[
S/3L(\{HL\}^6H8LH(LH)^6L)[(HL)^7H8LH(LH)^7L]^2H(LH)^8LH(LH)^5L'L'/air,
\]

where S represents the substrate that is either made of OHARA glass, WMS-02, with a refractive index of 1.658, or F7, with a refractive index of 1.606, both with high coefficients of thermal expansion. In this study Nb₂O₅ and SiO₂ are used as the high and low refractive-index materials, respectively, whose values of refractive index were 2.32 and 1.46 at λ=1550 nm, respectively. Here H and L denote the quarter-wave layers with high and low refractive indices, respectively. The last two layers, H’ and L’, are designed for antireflection to match the air ambient. Note that the layer sequence of the multilayer structure for the NBPF scheme is in terms of H and L, except the outmost H’ and L’ layers, and is symmetric so that the spectra farther away from the monitoring position will not be rapidly degraded during the coating process.

B. Coating process

The coating system mainly contains two rotational offset electron guns and a 16 cm Kauffman-type ion source. Moreover, oxygen is introduced as a working gas. The coating materials, Nb₂O₅ and SiO₂, are placed on the circular hearths in the two electron guns. The vacuum chamber is pumped down to a base lower than 10⁻⁴ Pa and is heated to 200 °C, permanent magnets are arranged around the ion source to supply magnetic fields in order to confine the plasma with a high degree of ionization. The mass flow controller is set to 15 SCCM (SCCM denotes cubic centimeter per minute at STP), and the working pressure is 1.8×10⁻² Pa. The substrate is 95 mm in diameter and 1.5 mm in thickness, and it is loaded on a holder rotating at 800 rpm. The high rotation rate ensures symmetrical film deposition.

The as-deposited films are then uniformly deposited throughout the substrate under stable deposition and etching rates for the two coating materials to achieve the lowest thickness variations that are necessary conditions for the 187 layer coating of the NBPF.

During the deposition process, an optical thickness monitor set at a wavelength of 1563.6 nm is used. The optical thickness of the NBPF films is monitored by using the turning-point method. Precise prediction of the turning points for the overall as-deposited layers is crucial for NBPF performance. Moreover, to increase the usable ring area of NBPF for mass production, the monitoring position is usually set at 7 mm off the center of the substrate. Consequently, a stable dielectric multilayer stack, with alternating Nb₂O₅ and SiO₂ thin films, with low stress, high packing density, high refractive index, low extinction, and low surface roughness, can be obtained.

C. Narrow bandpass filter measurement

The measurement system for DWDM filters consists of an HP 81632A power sensor module, an HP 81640A tunable laser, and an HP 8164A light-wave measurement system. The wavelength for the measurement ranges from 1500 to 1640 nm. The accuracy of the absolute wavelength is
plotted for 

\[ \eta = \frac{Y N_\delta \cos \theta_\delta}{C \cos \theta_\delta} \quad \text{for } s \text{ polarization (TE)}, \]  
\[ \eta_p = \frac{Y N_\delta \cos \theta_\delta}{C \cos \theta_\delta} \quad \text{for } p \text{ polarization (TM)}, \]  
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If \( \theta_p \), the angle of incidence, is given, the values of \( \theta_k \) and \( \theta_S \) can be found from Snell’s law, i.e.,

\[ N_0 \sin \theta_0 = N_k \sin \theta_k = N_S \sin \theta_S. \]  

The reflectance \( R \), transmittance \( T \), and absorbance \( A \) of the optical thin film structure can be obtained through the following equations:7

\[ R = \left( \frac{\eta B - C}{\eta B - C} \right) \left( \frac{\eta B - C}{\eta B + C} \right)^*, \]  
\[ T = \frac{4 \eta \Re(\eta)}{(\eta B + C)(\eta B + C)}, \]  
\[ A = \frac{4 \eta \Re(B C^* - \eta)}{(\eta B + C)(\eta B + C)}. \]  

C. Center wavelength shift and transmission curve distortion

For two or multiple cavity filters, which are composed of two or more of the Fabry-Pérot filters in series with the repetition number for periodic stacks \( m \), it has been extensively reported that the variation of optical path length at each film layer plays a dominant role on the change of transmission characteristics which mainly include the center wavelength shift and degradation of transmission curve. The variation of optical path length, \( \Delta(n d_i), i = H, L, \) can be generally expressed as

\[ \Delta(n d) = d \Delta n + n \Delta d = d \left( \frac{\partial n}{\partial T} \right)_\sigma \Delta T + \left( \frac{\partial n}{\partial \sigma} \right)_T \Delta \sigma \]  
\[ + n \left( \frac{\partial d}{\partial T} \right)_\sigma \Delta T + \left( \frac{\partial d}{\partial \sigma} \right)_T \Delta \sigma. \]  

It means that the variation of optical path length may be resulted from the change of refractive index \( n \) and the thickness of film layer \( d \) due to the effects of temperature \( T \) and stress \( \sigma \). If the variation of optical path length is identical not only at all low index layers but also at all high index layers, respectively, it will lead to center wavelength shift only without degradation of transmission curve. Otherwise, both center wavelength shift and transmission curve distortion will appear simultaneously.

If the variation of optical path length at high and low layers are identical, the ratio of center wavelength shift to center wavelength can be simplified to the form2

\[ 1 - \frac{\Delta L}{\lambda} \approx \frac{d}{\lambda}. \]
\[
\frac{\Delta \lambda}{\lambda} = \frac{R \Delta(n_H d_H) + S \Delta(n_L d_L)}{R[n_p d_H - \Delta(n_H d_H)] + S[n_L d_L - \Delta(n_L d_L)]},
\]
where \(L\) and \(H\) indicate the low and high layers, respectively, and
\[
R = \frac{(1 - r) + n_H(1 - s + 1)}{n_H(1 - r)} - \frac{n_H(1 - s + 1)}{(1 - s)},
\]
\[
S = \frac{r(1 - s + 1)}{n_L(1 - r)} - \frac{n_s(1 - s + 1)}{(1 - s)},
\]
\[
r = \left(\frac{n_L}{n_H}\right)^2, \quad s = \left(\frac{n_H}{n_L}\right)^2.
\]
Center wavelength shift of DWDM, therefore, may be resulted from the following factors.

1. Effects of temperature on optical index and film thickness

Influence of temperature change on refractive index, called thermo-optic effect, is determined by the thermo-optic coefficient of film material. It is presented in all practically used waveguide materials. Thermo-optic control of optical waveguide devices is attractive from viewpoint of simplicity and flexibility. Moreover, thermo-optic space switches are commercially available. The change in refractive index \(n\) of a material with temperature \(T\) is due to the change in density \(\rho\) and due to the temperature change itself. In fused silica, the thermo-optic effect is mainly due to the latter, which originates from the thermal changes in the polarizability. Materials that exhibit strong nonlinearity of refractive index or a thermo-optic effect are promising candidates for applications in optical switching devices. On the other hand, the influence of temperature change on the film thickness is determined by the thermal expansion coefficient of film material.

Influence of a temperature rise on the optical behavior of a multilayer film narrow bandpass filter has been investigated by the literature.\(^1\)\(^-\)\(^3\) Since the variation of optical path length at each high or low layer is almost identical due to a uniform temperature rise or drop, it, generally, only results in the center wavelength shift without serious degradation of transmission curve.

2. Effects of stresses on optical index and film thickness

The permittivity and dielectric constant, and hence the refractive index, are, in general, functions not only of the applied electric field but also of the stress on the material. The change of the refractive index caused by stress is called the photoelastic effect.\(^8\) The refractive index of a crystal is specified by the indicatrix, which is an ellipsoid whose coefficients are the components of the relative dielectric impermeability tensor \(B_{ij}\) at optical frequencies, namely,
\[
B_{ij}x_jx_j = 1,
\]
where the tensors \(B_{ij}\) is defined as \(B_{ij} = \kappa_{ij} \partial E_i / \partial D_j\).

FIG. 1. (Color online) Shift of center wavelength under different temperatures for NBPF with F7 substrate.

Thus the small change of refractive index produced by stress is a small change in the shape, size, and orientation of the indicatrix. This change is specified by giving the small changes in the coefficients \(B_{ij}\).

If we neglect higher-order terms than the first in the field of stresses, the changes \(\Delta B_{ij}\) in the coefficients are given by
\[
\Delta B_{ij} = \phi_{ijkl} \sigma_{kl} \quad \text{or} \quad \Delta B_{ij} = p_{ijrs} \varepsilon_{rs},
\]
where \(\phi_{ijkl}\) and \(p_{ijrs}\) are called the piezo-optical and strain-optical coefficients, typically having the orders of magnitude of \(10^{-12}\) Pa\(^{-1}\) and \(10^{-1}\), respectively.

By using the relation, \(B = 1/n^2\), it is assumed that the change of refractive index for an isotropic film material can be written as\(^8\)\(^9\)
\[
\frac{\partial n}{\partial \sigma} = -\frac{1}{2}n^3 \varphi.
\]

Consequently, the optical performance of optical thin film may be changed due to the alteration of refractive index induced by film stress, as shown in Eq. (13). On the other hand, the change rate of the \(i\)th layer thickness due to stresses can be easily obtained through the following equation:
\[
\left[\frac{\partial d_i}{\partial \sigma_{ij}}\right]_T = -\frac{2v_i d_i}{E_i(1 - v_i)}.
\]

Based on the above equations, it clearly appears that if the stress level is identical not only at each high layer but also at each low layer, then the consistency in the variation of optical path length will lead to only center wavelength shift instead of degradation of transmission curve. In this report, an accurate mechanical model is adopted to calculate the distributions of film stress. Moreover, the influence of stresses on the transmission characteristics is evaluated and discussed.
Five-cavity subnanometer bandwidth filter of the design, $S/3L(HL)^6\frac{H}{8L}H(LH)^6L[(HL)^7H8LH(LH)^7L]^5H(LH)^68LH(LH)5L'H'\text{air}$, is adopted in the analysis. The total number of film layers, therefore, is 187. The materials of high and low refractive indices are Nb$_2$O$_5$ and SiO$_2$, respectively. The central wavelength shifts of NBPFs, individually deposited on F7 and WMS-02 substrates, under different temperature rises are measured and plotted, as shown by the dots in Figs. 1 and 2, respectively. It is interesting to find that there exist different shifts for these two NBPFs deposited on different substrates. The NBPF of F7 presents a redshift, while the NBPF of WMS-02, on the contrary, reveals a blueshift as the temperature increases. These experimental data can be approximately represented by the linear relations $\Delta \lambda = 0.0013\Delta T + 1549.8$ and $\Delta \lambda = -0.0008\Delta T + 1541.8$, respectively.

The thermal stresses induced in the thin films due to temperature rise are evaluated by improved laminate model and shown in Table II, where the $x$ axis is parallel to the plane of thin film layers. In this study, only the stress-optical coefficient, $(\partial n/\partial \sigma)_T$, and thermal-optical coefficient, $(\partial n/\partial T)_x$, for dielectric oxide film materials, such as TiO$_2$, Ta$_2$O$_5$, Nb$_2$O$_5$, and SiO$_2$, have not been sufficiently clarified. According to the limited data available at hand, the magnitudes of $(\partial n/\partial \sigma)_T$ and $(\partial n/\partial T)_x$, for the material SiO$_2$ are equal to $-3.53 \times 10^{-12}$ Pa$^{-1}$ (Ref. 9) and $1.18 \times 10^{-5}$ $\text{C}^{-1}$, respectively. The corresponding data for Nb$_2$O$_5$ film material, however, are still absent in the literature and will be determined in this article.

The thermal stresses induced in the thin films due to temperature rise are evaluated by improved laminate model and shown in Table II, where the $x$ axis is parallel to the plane of thin film layers. In this study, only the stress-optical coefficient, $(\partial n/\partial \sigma)_T$, and thermal-optical coefficient, $(\partial n/\partial T)_x$, of the film material, Nb$_2$O$_5$, are unknown properties. These two properties can be easily evaluated inversely and are given in Table III. It has been reported that the value of $\Delta n/\Delta \sigma$, similar to the definition of the stress-optical coefficient, for a number of Nb$_2$O$_5$ film samples deposited by both dc and MF magnetron sputtering processes under various process parameters is confined within the range from $-100 \times 10^{-12}$ to $-800 \times 10^{-12}$ Pa$^{-1}$. Since the index of refraction of a material is correlated to its density, it can be assumed that with an increased particle bombardment in the film deposition process the associated densification process will yield less level

**TABLE I.** Material properties of film layers and substrate (Refs. 10–14).

<table>
<thead>
<tr>
<th>Material</th>
<th>Young’s modulus (GPa)</th>
<th>Coefficient of thermal expansion (ppm $\degree$C$^{-1}$)</th>
<th>Optical refractive index</th>
<th>Poisson’s ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Film Nb$_2$O$_5$</td>
<td>60</td>
<td>5.8</td>
<td>2.32</td>
<td>0.2</td>
</tr>
<tr>
<td>Film SiO$_2$</td>
<td>74.5</td>
<td>0.55</td>
<td>1.46</td>
<td>0.164</td>
</tr>
<tr>
<td>Substrate</td>
<td>85</td>
<td>10.1 $+$ 0.015($T-20$)</td>
<td>1.569</td>
<td>0.2</td>
</tr>
<tr>
<td>WMS-02</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Substrate</td>
<td>55</td>
<td>9.8 $+$ 0.006($T-20$)</td>
<td>1.606</td>
<td>0.2</td>
</tr>
<tr>
<td>F7</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**TABLE II.** Thermal stress $\sigma_{xx}$ induced in thin films due to temperature rise $\Delta T=70$ $\degree$C for two different NBPFs.

<table>
<thead>
<tr>
<th>Substrate of NBPF</th>
<th>Thermal stress $\sigma_{xx}$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>WMS-02</td>
<td>22.7–22.9</td>
</tr>
<tr>
<td>F-7</td>
<td>19.2–19.5</td>
</tr>
</tbody>
</table>

**TABLE III.** The stress-optical and thermal-optical coefficients of film material Nb$_2$O$_5$.

<table>
<thead>
<tr>
<th>Film material</th>
<th>Thermal-optical coefficient $(\partial n/\partial T)_x$</th>
<th>Stress-optical coefficient $(\partial n/\partial \sigma)_T$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nb$_2$O$_5$</td>
<td>$1.43 \times 10^{-5}$ $\text{C}^{-1}$</td>
<td>$-95.1 \times 10^{-12}$ Pa$^{-1}$</td>
</tr>
</tbody>
</table>
of porosity and higher values of the refraction index. Moreover, it is obvious that for the same material a less level of porosity will correspond to a smaller value of the stress-optical coefficient. Compared to previous dual magnetron sputtering process, since a less level of porosity or denser film material can be achieved by IAD process, a smaller magnitude of the stress-optical coefficients of Nb$_2$O$_5$ thin film material are evaluated by measuring the data of central wavelength shifts due to a temperature rise. These coefficients are evaluated and approximately equal to $-95.1 \times 10^{-12} \text{ Pa}^{-1}$ and $1.43 \times 10^{-5} \text{ °C}^{-1}$, respectively.

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TABLE IV. Contributions of the change of refractive index and film thickness due to temperature rise ($\Delta T=70^\circ \text{C}$) and thermal stress on shift of center wavelength.

<table>
<thead>
<tr>
<th>Substrate</th>
<th>$\Delta \lambda$ due to $\partial d / \partial T$ (nm)</th>
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<th>$\Delta \lambda$ due to $\partial n / \partial T$ (nm)</th>
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<td>F7</td>
<td>0.344</td>
<td>-0.302</td>
<td>0.772</td>
<td>-0.723</td>
<td>0.091</td>
</tr>
<tr>
<td>WMS-02</td>
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<td>-0.334</td>
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**V. CONCLUDING REMARKS**

In this article, two five-cavity thin film NBPFs are fabricated by using the same Nb$_2$O$_5$ and SiO$_2$ film materials individually deposited on different substrates, F7 and WMS-02, by IAD process. It is observed that the NBPF of F7 presents a redshift, while the NBPF of WMS-02, on the contrary, reveals a blueshift as the temperature increases. The mechanism and theory related to this central wavelength shift due to temperature change are investigated and discussed. It is found that the different levels of thermal stress induced in the thin films of two NBPFs play a key role on the final situation of central wavelength shift. Moreover, the magnitudes of the stress-optical and the thermal-optical coefficients of Nb$_2$O$_5$ thin film material are evaluated by measuring the data of central wavelength shifts due to a temperature rise. These coefficients are evaluated and approximately equal to $-95.1 \times 10^{-12} \text{ Pa}^{-1}$ and $1.43 \times 10^{-5} \text{ °C}^{-1}$, respectively.

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