Laser Original

Optical Waveguide Characterization of Thin Films

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Guided wave techniques are powerful to study the properties of different optical coatings. The latest results are presented to illustrate the interest of these techniques: the dependence on ion energy of the anisotropy of ion assisted TiO₂ films is given; the measurement of losses during propagation permits to study the origin of losses in multilayer coatings and in particular to demonstrate that absorbing transition layers have to be introduced in the model; properties of ion implanted Ta₂O₅ layers are also investigated. The refractive index profile of Ta₂O₅ layers implanted with Ti is determined by using the *m*-line technique; coupling a pump beam at 488 nm in different guided modes, the photoluminescent properties around 1.53 μm of Er implanted Ta₂O₅ layers are also shown.

Key Words: Optical thin films, Guided wave, Anisotropy, Ion implantation, Photoluminescence.

1. Introduction

Powerful characterization tools are necessary to make high quality thin films for high performance optical systems. Techniques based on the excitation of waves which are guided in the thickness of the coatings are very interesting to investigate the optical properties of thin films¹⁾. Because a guided mode corresponds to a resonant state of the optical wave and because the interaction length between light and the material is important compared to the wavelength, the conditions of propagation are very sensitive to the optical properties of film materials. The refractive index n , the thickness t of layers and the optical losses can be accurately measured with these techniques as well as their modification with temperature²⁾ or under light illumination³⁾.

After briefly recalling the principles of these

techniques we present here a summary of recent results obtained for single layers as well as for a few multilayers.

2. Principles of guided wave techniques

2.1 *m*-line technique

The determination of guided mode propagation constants in a planar waveguide by the *m*-line technique (Fig. 1) consists in measuring the angles for which dark lines appear in a beam reflected on a prism coupled to the waveguide⁴⁾. The propagation constants of the guided modes can then be determined from these angles. From the propagation constants of two guided modes we can obtain independently the n and t of a homogeneous and isotropic single layer. The uncertainties on n and t of such a homogeneous layer are $\sim 1 \times 10^{-3}$ and ~ 1 nm, respectively.

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Fig. 1 *m*-line technique

2.2 Prism coupler

If the light is incident close to the edge of a prism, it can be coupled in the guide and not coupled back in the prism. The losses in coatings can be studied by recording the image of the light that has been coupled out of the guide as a result of scattering along the guided path. For this we use a conventional digital imaging system⁵⁾. The signal is filtered and approximated by an exponential law. The method is very sensitive and losses as low as 1 dB/cm can

be measured. When modeled as simple absorption, such losses correspond to an extinction coefficient k of the order of 1×10^{-6} for a single layer. This method works up to 80 dB/cm corresponding to a k value on the order of 1×10^{-4} .

3. Some results of characterization

Different results obtained from guided wave characterization techniques have been presented in refs. 1 to 3. We give here some recent results.

3.1 Dependence of the anisotropy of ion assisted deposited TiO_2 films on ion energy.

TiO_2 layers obtained by Ion Assisted Deposition⁶⁾ (IAD) can be quite isotropic when using a 250 eV beam of O_2 . It is no longer the case when using an ion beam of lower energy. We are here concerned with the dependence of this anisotropy on the ion energy. The film anisotropy is studied by using both the propagation constants measured with the *m*-line technique and with cross-polarization transmission measurements⁷⁾. A biaxial model⁸⁾ associated with the columnar structure⁹⁾ is used. The principal refractive index n_3 is taken in the direction of the columns; the other two, n_1 and n_2 , form a direct reference with n_3 .

Deposition conditions

TiO_2 films studied were obtained by IAD in a Balzers BAK 750 equipped with a cryogenic pump. The background pressure is 1×10^{-6} hPa. An argon pressure of 5×10^{-5} hPa is introduced through the ion gun (8 cm Kaufman-type ion gun) and a total pressure of 1.5×10^{-4} hPa is maintained by adding O_2 through the ion gun. The starting material is TiO_2 tablets and the deposition rate of 0.2 nm/s is regulated with a quartz crystal. The substrates (BK7 glass) do not rotate during deposition. The substrate temperature is not controlled and is less than 100 °C. The vapor incidence is 20.3° on all the studied samples. The ion beam is centered on the substrate and the angle of incidence of the ions on the substrate is 23°. The distance between the output grid of the ion gun and the substrate is 400 mm. The ion current density is approximately $230 \mu\text{A}/\text{cm}^2$ on the substrate.

Figure 2 gives the dependence with the ion energy of the differences $n_3 - n_1$ and $n_2 - n_1$ measured in air. The thickness of the films are of

Fig. 2 Dependence of $\Delta n_{31} = n_3 - n_1$ and $\Delta n_{21} = n_2 - n_1$ on ion energy

~ 700 nm. So we show that the anisotropy of TiO_2 films can be varied when using IAD by changing the ion energy. This result is in good agreement with ref. 10.

3.2 Ion implanted Ta_2O_5 layers

Ion implantation largely used in microelectronics can be employed to introduce metals in optical thin films. Different materials have been implanted in Ta_2O_5 layers to modify their optical properties. After implantation the layers are strongly absorbing but after baking in air for 48 h at 400 °C they recover their high transparency. With ion implantation and depending on the fluence the layers become inhomogeneous. The guided modes are significantly modified by the implantation. The *m*-line technique has been used to determine the refractive index profile in the thickness of these implanted layers. An example of such a profile is given in Fig. 3. It concerns a Ta_2O_5 layer deposited on a silica substrate by ion plating¹¹⁾ implanted with 2×10^{16} Ti ions/ cm^2 at 360 keV. The losses with propagation of TE_0 guided mode at 632.8 nm are only 2dB/cm as before as well as after the implantation. As implantation can be performed through masks it should be a good way to increase the refractive index locally, keeping losses very low.

Fig. 3 Refractive index profile of Ti implanted Ta_2O_5 layer (2×10^{16} at/cm², 360 keV) after baking in air at 400°C for 48h; a) at 514.5 nm; b) at 632.8 nm.

3.3 Refractive index of layers embedded in a multilayer stack.

The *m*-line technique can also be used to determine the refractive index and the thickness of layers composing a multilayer stack. In a multilayer the intensity distribution strongly depends on the guided mode considered, more than for a single layer. This of course varies with the composition of the guide. If there are less modes than parameters (n and t for each layer) one needs to use simplifying hypothesis. It can be assumed that the n of layers composed of a given material are the same whatever the positioning of the layer; the optical thickness can be supposed to be known if accurate optical monitoring is used during deposition. But the structure of the stack can be chosen to have several

guided modes in such a way that we can have enough information. This composition can also be chosen to have some modes relevant on the properties of specific layers. It then becomes a powerful tool to investigate the behavior of films in stratified media.

We present here the example of a study we performed on a multilayer stack. The structure considered is silica substrate/*HL 8H LH*/air where *H* and *L* represent layers of optical thickness $\lambda_1/4$ ($\lambda_1 = 632.8$ nm) of high refractive index (Ta_2O_5) and low refractive index (SiO_2), respectively. The layers have been deposited by ion plating. The refractive index and the thickness of the layers, both determined for three wavelengths (632.8 nm, 514.5 nm and 488 nm) are given in Table I. The effective index calculated with the values of Table I are the same as those measured within 1×10^{-4} . So, as was confirmed by studies of other structures the Ta_2O_5 layers and the SiO_2 layers deposited by ion plating have a refractive index which does not depend on the position in the stack. The thickness of each layer is found to vary by 1.3 % at the most from one wavelength to another. This example shows that the method is reliable and can be used to study coatings for lasers.

3.4 Origin of the losses in a multilayer structure

It is important to know the origin of losses in optical coatings, in particular for high power lasers. It can be absorption or scattering from the volume of the materials or from the boundaries. To perform such a study one needs to make different measurements as scattering¹²⁾ and photothermal deflection¹³⁾. The measurement of losses with guided propagation is complementary and permits one to trace their origins. As presented in the previous part, depending on the design of the stack and on the mode

Table I Refractive index and thickness of the layers composing the structure silica substrate/*HL 8H LH*/air where *H* and *L* represent layers of optical thickness $\lambda_0/4$ ($\lambda_1 = 632.8$ nm) of high refractive index (Ta_2O_5) and low refractive index (SiO_2), respectively.

	$\lambda = 632.8$ nm	$\lambda = 514.5$ nm	$\lambda = 488.0$ nm
n_H (layer #1)	2.223	2.265	2.278
n_L (layer #2)	1.480	1.503	1.504
n_H (layer #3)	2.223	2.265	2.278
n_L (layer #4)	1.480	1.503	1.504
n_H (layer #5)	2.223	2.265	2.278
t_H (layer #1)	71.0	70.2	70.2
t_L (layer #2)	111.3	110.8	110.1
t_H (layer #3)	568.5	561.1	562.4
t_L (layer #4)	111.3	110.7	110.7
t_H (layer #5)	71.0	70.1	70.4

considered, the guided light can have most of its energy in one layer or another or it can be spread all over the stack. Consequently the losses also depend on the mode order.

Consider a coating composed of silica/*4H L 5H L 6H*/air where *H* and *L* denotes layers of $\lambda_0/4$ optical thickness for high refractive index material (2.45) and low refractive index material (1.49), respectively. This structure can support 7 guided modes for TE polarization and 6 for TM. Fig. 4 gives the intensity distribution for the mode orders 0 to 4 for both polarization states. It can clearly be seen that TE_0 , TE_3 and TM_0 modes are concentrated mainly in the *6H* layer close to the air; TE_1 , TE_4 and TM_1 modes are mainly concentrated in the *5H* layer which is in the middle of the stack whereas TE_2 and TM_2 modes are concentrated mainly in the *4H* layer close to the substrate. Moreover an important part of the guided energy is located close to the interfaces for TM modes -particularly for TM_3 and TM_4 modes-. This structure has been chosen to have these particularities. Assuming there is only volume absorption the losses can be calculated from the imaginary parts of the

propagation constants. Such a model fails to explain the dispersion of the losses for different guided modes.

In ref. 14 the authors show that absorbing transition layers should be considered to explain photothermal deflection measurements performed on Ta_2O_5 made by ion plating.

A good agreement between the measured and calculated values is obtained with such a hypothesis of absorbing transition layers at each layer boundaries. Table II and III give the model used for calculation and the calculated and the measured losses for the mode orders 0 to 4, respectively. Nearly all the measured losses are then explained. Of course other explanations could be given. In particular it would be possible to decrease the transition layer thicknesses and to increase the extinction coefficients. But in any case one has to consider absorbing transition layers. Also taking into account scattering at the interfaces could again give a better explanation of the results. The study has been performed for $\lambda_0 = 514.5$ nm ; the substrate was a well polished silica substrate (a few Angstroms rms roughness).

Fig. 4 Intensity distribution for the guided modes propagating for both polarization states in the 5 layer guide air/6H L 5H L 4H/substrate where H and L denote $\lambda_0/4$ layers of high and low refractive index, respectively ($\lambda_0 = 514.5$ nm). a) modes TE_0 , TE_1 and TE_2 ; b) modes TE_3 and TE_4 ; c) modes TM_0 , TM_1 and TM_2 ; d) modes TM_3 and TM_4 .

Table II Model used to calculate the losses during guided propagations consecutive to absorption in the 5 layer guide air/6H L 5H L 4H/substrate where H and L denote $\lambda_0/4$ layers of high and low refractive index, respectively ($\lambda_0 = 514.5$ nm). k is the extinction coefficient ($\lambda_0 = 514.5$ nm).

material	0.01 silica	0.1 H	3.9 H	0.1 L	0.9 L	0.1 H
d[nm]	1	6	222	9	78	6
k	10^{-3}	$2 \cdot 10^{-4}$	$4 \cdot 10^{-6}$	$8 \cdot 10^{-5}$	$3 \cdot 10^{-5}$	$7 \cdot 10^{-4}$
material	4.9 H	0.1 L	0.9 L	5.8 H	0.2 H	
d[nm]	279	9	78	330	11	
k	10^{-6}	10^{-4}	$8 \cdot 10^{-6}$	$2 \cdot 10^{-6}$	$3.5 \cdot 10^{-4}$	

Table III Losses calculated and measured during guided propagation in the 5 layer guide air/6H L 5H L 4H/ substrate defined in Table III ($\lambda_0=514.5$ nm).

mode	losses [dB/cm]		mode	losses [dB/cm]	
	calc.	measu.		calc.	measu.
TE ₀	4.6	4.9 ± 1	TM ₀	5.0	3 ± 1
TE ₁	6.9	6.4 ± 0.3	TM ₁	8.2	5.5 ± 2
TE ₂	7.3	7.4 ± 0.3	TM ₂	11.4	11 ± 1.5
TE ₃	13.1	12.5 ± 2	TM ₃	14.8	14.1 ± 0.5
TE ₄	22.0	26.5 ± 3	TM ₄	23.8	26 ± 2

3.5 Photoluminescent properties of Er implanted layers

One can also take advantage of the propagation of a guided wave to study Raman scattering or the photoluminescent properties¹⁵⁾ of the material composing the guide. Optical planar waveguides doped with rare-earth ions offer numerous attractive features to be used as amplifiers or lasers in optical communications. When incorporated in a trivalent state into a solid host, these ions exhibit relatively sharp optical transitions corresponding to intra-4f states. Erbium is of particular interest because of its intra-4f transition with a wavelength emission around $1.54 \mu\text{m}$, coinciding with the low-loss window of standard optical telecommunications silica fibers.

Tantalum pentoxide (Ta_2O_5) films of optical thickness $6\lambda_1/4$ ($\lambda_1=0.6328 \mu\text{m}$, $n=2.2$) are deposited on fused silica substrates by ion plating. The Er ions are incorporated into the waveguides by ion implantation. The implantation fluences range from 3.3×10^{14} to 2×10^{15} ions/cm² and the energy ranges from 190 keV to 380 keV. Post implantation thermal annealing is carried out on the samples at 400 °C for 10 hours.

As for Ti or Li implanted layers, Er implanted waveguides exhibit losses on guided propagation which are as low as the losses in the

Fig. 5 Fluorescence spectra from a Ta_2O_5 film implanted with 0.5 at.% of Erbium (380 keV- 1.7×10^{15} ions/cm²). Pump beam: TE₁ mode at $\lambda=488$ nm.

non implanted ones (a few dB/cm for $\lambda_1=632.8$ nm).

The experimental apparatus used to record the fluorescence spectra is similar to the one used to study the losses on guided propagation¹⁾.

Figure 5 gives an example of the fluorescence spectrum of a waveguide with an Er ion concentration of 0.5 % (380 keV 1.7×10^{14} ions/cm²). This spectrum is obtained when coupling the pump beam at $\lambda=488$ nm in the mode TE₁ of the waveguide. The pump beam is coupled with a 70 % efficiency. Such efficiency is determined from the near field shape on the prism base³⁾.

Fig. 6 Fluorescence spectrum for the sample of Figure 5. Pump beam: TE_0 mode at $\lambda = 488$ nm.

We note that this spectrum is peaked at $\lambda = 1.53 \mu\text{m}$, which coincides¹⁶⁾ to the wavelength of the transitions between the first excited manifold $^4I_{13/2}$ and the $^4I_{15/2}$ ground manifold of Er^{3+} .

Figure 6 shows the fluorescence spectrum in the same conditions as the one corresponding to Fig. 5 (Er ion concentration 0.5 %) but with the pump beam coupled in the TE_0 mode of the waveguide ($\lambda = 488$ nm). The coupling efficiency is also 70 %. The spectrum shape is quite the same as in Fig. 5 with a peak intensity around $\lambda = 1.53 \mu\text{m}$ while the intensity is approximately divided by two. Fig. 7 gives the intensity of the pump field for (A) TE_0 and (B) TE_1 modes. These intensities are normalized in order to obtain the same guided power flow for both modes. The hatched region, located around 50 nm under the surface, indicates the location of the Er ions (energy: 380 keV, SIMS analysis). From a simple integration of the pump energy in the dashed region it appears that coupling the pump beam in TE_1 mode is 2.5 times as efficient as coupling it in the TE_0 mode. Indeed we find a fluorescence intensity twice as great when the

(a)

(b)

Fig. 7 Normalized intensity of the pump field for (A) TE_0 and (B) TE_1 modes. The hashed region indicates the location of the Er ions.

guide is pumped with the TE_1 mode instead of the TE_0 mode. The slight discrepancy between the expected factor (2.5) and the measured one (2) is probably due to the fact that the TE_1 mode is over estimated because absorption has not been taken into account in the calculations used in Fig. 7.

4. Summary

Guided wave techniques are particularly use-

ful to study the intimate properties of thin films.

Because they can be used to determine the refractive index of layers, their anisotropy, the refractive index profile in the thickness, the origin of losses these techniques keep yielding new results.

Recently they have been employed to study layers in multilayer stacks; they confirm the presence of absorbing transition layers on the films' boundaries; besides, guided wave techniques appear to be particularly effective to study ion implanted layers and the luminescence of erbium doped layers which could be attractive to produce integrated devices such as amplifiers or lasers for optical communications.

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