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Pulsed Laser Deposition of Er doped tellurite films on large area

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Abstract Thin films of Er^{3+} -doped tungsten tellurite glass have been prepared by the pulsed laser deposition technique using an ArF excimer laser. The depositions were performed at different O₂ pressure (5, 10 Pa) and at different substrate temperatures (RT, 100°C and 200°C). The composition and the optical properties of the deposited films, such as transmission, dispersion curves of refraction index and extinction coefficient, and film thickness were studied for the different deposition parameters. Transparent films at the highest substrate temperature were obtained only for a higher oxygen pressure with respect to the RT conditions.

1. Introduction

In recent years, due to the rapid increase of information capacity and the need for flexible networks, there exists urgent demand for optical amplifier with a wide and flat gain spectrum to be used in wavelength–division–multiplexing (WDM) network systems [1]. To achieve the amplification of optical signals in the third telecommunications window, the ${}^{4}I_{13/2} \rightarrow {}^{4}I_{15/2}$ transition of $\mathrm{Er}^{3^{+}}$ ions at 1.5 µm can be used. So far, much effort has been spent to find good candidates to substitute or complement the typically used silicabased matrix host. Tellurite glasses appear promising materials as they combine an ${}^{4}I_{13/2} \rightarrow {}^{4}I_{15/2}$ emission bandwidth as broad as 60 nm and extended up to 1630 nm with acceptable features for fiber drawing and planar waveguide fabrication [2-4]. Furthermore, they exhibit a wide transmission range (0.35 to 5 µm), the lowest vibrational energy (about 780 cm⁻¹) among oxide glass formers, low process temperature, good chemical stability and non linear optical properties [5]. Recently, some attention has been devoted to tungsten tellurite glasses [6].

Several methods have been attempted to produce tellurite waveguides, such as sol-gel, ion-exchange, and sputtering [7-9]. However, the fabrication of good quality erbium-doped tellurite waveguides is still a challenge. Pulsed laser deposition (PLD) is very attractive, since it is an excellent technique to produce complex oxide thin films. Here we study the deposition of tellurite films of complex stochiometry on substrates kept at different temperatures. It is well known that the transparency of PLD deposited oxide films strongly depends on the oxygen content in the layers: understoichiometric films are less transparent [10, 11]. To avoid this feature, a background molecular oxygen pressure is necessary during deposition.

The aim of this work is to obtain transparent tellurite films by PLD and to understand the influence of substrate temperature in the deposition process. The depositions were performed in order to obtain quite uniform films on a large area, because this feature is very important for the development of planar waveguides.

2. Experimental

Tellurite films have been produced starting from a tungsten tellurite glass of nominal composition 45%TeO₂--39%WO₃--15%Na₂O--1%ErO₃. The used experimental setup consists of an ArF excimer laser (λ =193 nm, τ =20 ns), a stainless steel chamber and a rotative-turbo molecular pump system (residual pressure $3x10^{-5}$ Pa). The laser beam was directed to the target at the angle of 45° ; the laser fluence was about 2 J/cm². The substrate was mounted on an x-y movimentation system in order to deposit a uniform thick layer on an area of about 4 cm².

The films were deposited at three different substrate temperatures (RT, 100°C and 200°C) on pure silica (from Hereaus) and on crystalline silicon substrates in a background pressure of molecular oxygen (5 and 10 Pa). The distance between the target and the substrate was 55 mm when operating at room temperature, and 45 mm for depositions performed at 100°C and 200°C. A total number of 40000 laser pulses was used to obtain 1.6 μ m thick layers on silica substrates. Thinner films were deposited on silicon substrates to allowed a non-ambiguous identification and quantitative determination of the different elements present in the films by Rutherford backscattering spectroscopy (RBS) using a ⁴He⁺ beam at 2.2 MeV.

The optical transmission spectra of the films were acquired by using a double beam spectrophotometer Perkin-Elmer Lambda 900, in the NIR-Visible-UV regions (200-2500 nm). The spectra were used as input for a computer code in order to obtain the dispersion curves of the refraction index n, extinction coefficient k and the film thickness.

3. Result and discussion

Figure 1 shows the transmittance spectra of the films deposited at increasing substrate temperature (RT, 100° C and 200° C), at the same O₂ pressure (5 Pa). The films transmittance decreases as the substrate temperature increases. The oscillations observed in the spectra are typical fringes due to interference between the light reflected at the air-film and film-substrate interfaces.

Using the transmission spectra as input, a computer code (Refractor) [12] calculated the dispersion curves of the refractive index n and of the extinction coefficient k for the different films (Figures 2 and 3). Higher values of refractive index and extinction coefficient were determined for the films deposited at 100 $^{\circ}$ C and 200 $^{\circ}$ C with respect to the ones deposited at RT.



Figure 1. Transmission spectra of the films deposited at different substrate temperatures in 5 Pa of O_2 .



Figure 2. Refractive index of the films deposited at different substrate temperatures in 5 Pa of O_2 .

Figure 3. Extinction coefficient of the films deposited at different substrate temperatures in 5 Pa of O_2 .

10 Pa of O_2 ambient pressure was necessary to obtain a transparent film for deposition performed at the highest substrate temperature: the comparison between the films deposited at 200°C in different O_2 pressure is shown in figure 4.



Figure 4. Films deposited at 200 °C at 10 Pa of O_2 (dashed line) and at 5 Pa of O_2 (solid line).

To correlate the optical properties with the stoichiometry of the films, RBS analysis was performed and the experimental spectra were simulated with the RUMP computer program (Table 1). Films deposited at RT and at 100°C presented almost the same composition of the target. At the substrate temperature of 200°C two aspects become evident: on one hand the oxygen content of the film decreases, on the other, the relative concentration of tungsten atoms appears to increase.

Table 1. Composition of target and films as determined by RBS, error is 5%	%.
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Sample	%O	Te%	W%	Na%	Er%	
Target	66.2	13.2	11.5	8.8	0.3	
RT-5 $Pa(O_2)$	66.4	14.3	12.4	6.3	0.6	
100°C -5 Pa(O ₂)	66.4	14.3	12.4	6.3	0.6	
200°C -5 Pa(O ₂)	60.4	15.9	15.9	7.1	0.7	
200°C -10 Pa(O ₂)	62.4	14.9	14.9	6.6	0.7	

The results presented in this work show that not only the oxygen pressure but also the substrate temperature strongly influences both the optical and compositional properties of the films. The films deposited at RT in 5 Pa of oxygen are nearly stoichiometric respect to the target. They are very transparent and present values of the refractive index very close to the bulk target ones. This suggests that deposition at room temperature and at 5 Pa of O_2 pressure, enable the formation of a glass structure similar to the starting material. Films deposited at the same oxygen pressure but at higher temperatures are less transparent and present a lack of oxygen and a relative increase of W (probably because of metal segregation) as evidenced by RBS analysis. At highest temperature (200°C) the lack of oxygen is partially restored using a higher O_2 pressure (10 Pa) during the deposition. However, the film is very transparent and presents a refractive index value close to the target one. This result can be ascribed to the peculiarity of the deposition technique to form metastable phases.

4. Conclusion

PLD is a very promising technique to deposit tellurite film of complex composition. Transparent films, starting from a tungsten tellurite glass target, were obtained. Films were deposited on a large area with quite uniform thickness. This aspect is very important for optical application. During depositions at room temperature, an ambient pressure of 5 Pa of O_2 is sufficient to control the stoichiometry of the deposited layer. We show that the substrate temperature is a very critical parameter because it strongly influences the optical features and the composition of the films. Transparent films were also obtained at the highest temperature by increasing the oxygen pressure during depositions. The change of the content of one component (tungsten in our case) of the film by varying the substrate temperature, without changing the transparency and the refractive index of the films, is a very interesting feature that was never investigated so far at the best of our knowledge.

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