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Effects of hot-isostatic pressing and annealing post-treatment on HfO_2 and Ta_2O_5 films prepared by ion beam sputtering

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ABSTRACT

Hafnium oxide (HfO_2) films and Tantalum oxide (Ta_2O_5) films prepared by ion beam sputtering are important high refractive index materials from ultraviolet to near infrared. In this paper, Hot-isostatic pressing (HIP) technique is applied to the post-treatment of HfO_2 and Ta_2O_5 films, and the effects of HIP and annealing treatment on optical and stress properties of the thin films were compared. Optical properties are mainly including thickness, refractive index, extinction coefficient, band gap (E_g), and tail energy (E_u), which were calculated by the inversion of spectral properties. And stress characteristics were calculated by the curvature change of the samples surface. The results show that the two post-treatment methods both can reduce refractive index and extinction coefficient of the thin films, and can effectively improve band gap of thin films. The annealing treatment is better than HIP for reducing the extinction coefficient of thin films. The tail energies of the thin films show a decreasing trend after post-treatment, which indicates the decrease of the defect density of local state and the disorder degree of microstructures. The change of stress after HIP treatment is less than that of annealing because of the homogeneous pressure in HIP post-treatment, but both the post-treatment methods can transform the stress properties of the films.

1. Introduction

HfO_2 and Ta_2O_5 films are one of the most important high refractive index materials from the ultraviolet to near-infrared region. Ta_2O_5 films are the preferred high-refractive-index materials in the field of ultra-low loss laser films [1], and HfO_2 films are the preferred high refractive index materials in the field of high laser damage threshold films [2]. The main preparation methods of this two kinds of oxide films are electron beam evaporation, ion assisted deposition, ion beam sputtering, magnetron sputtering and other physical vapor deposition techniques. The metal oxide thin films prepared by ion beam sputtering have the advantages of small absorption, low scattering and low defects density [3–8]. This technique has become important method for preparing HfO_2 and Ta_2O_5 films and is widely used in optical multilayer element for laser optics [9].

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In the field of optical thin film technology, post-treatment is a commonly used thin film modification method [10–16]. Heat treatment can further reduce the stress and absorption characteristics of the thin film, and also improve the refractive index characteristics of the thin film. The heat treatment mainly includes UV-assisted heat treatment, vacuum heat treatment, heat treatment under specific atmosphere, rapid thermal annealing, especially the methods of heat treatment in vacuum treatment or aerobic atmosphere were widely reported and can greatly improve the performance of the thin film.

In the optical material technology, HIP post-treatment technique is widely used in zinc sulfide, zinc selenide and other infrared optical materials. Lewis et al. [17] reported that the visible light transmittance of zinc sulfide materials and zinc sulfide can increase from 10% to 50% using HIP treatment, and the transmittance can be also improved from middle wave infrared to long wave infrared. Biswas et al. [18] systematically investigated the effect of HIP on the crystalline transformation, microstructure, optical and mechanical properties of zinc sulfide materials. The application of HIP technique can further reduce grain boundaries, increase grain size and improve optical properties of materials.

In the field of optical thin films, HIP technique is rarely reported. In this paper, the effects of HIP treatment on HfO_2 and Ta_2O_5 films by ion beam sputtering were studied, and the effects of heat treatment on them were also discussed.

2. Experimental methods

2.1. Sample preparation

The metal oxide thin film was prepared using reoxidation method by ion beam sputtering deposition, and high purity metal target (target purity greater than 99.95%) was used as sputtering material. Ion source is the core of sputtering deposition technology, and 16 cm diameter radio frequency source was used as a sputter source. HfO_2 and Ta_2O_5 films were deposited on substrates of fused silica with various types. Samples with single-side polished substrates($\Phi 40 \times 6$ mm) were used to measure reflectance spectral, while double-side polished substrates were used to measure transmittance spectral. And single-side polished substrates with size of $\Phi 25 \times 1$ mm samples were used to measure the surface shape for calculating stress.

The preparation parameters of both oxide thin films were as follows: background vacuum was better than 1.0×10^{-3} Pa, ion beam voltage was 1250 V, ion beam current was 600 mA, oxygen flow rate was 40 sccm. The sputtering time of metal Hafnium target was 4850s and 9250s, and HfO_2 films were treated using atmospheric annealing and HIP methods respectively. The sputtering time of metal Tantalum target was 1800s and 2350s, and Ta_2O_5 films were also treated using the same two methods respectively.

The annealing treatment parameters were as follows: the temperature was set at 300 °C for 24 h, then naturally dropped to room temperature. The HIP treatment was treated by vacuum pressure and the treatment parameters were as follows: the background vacuum was 10 Pa, argon gas pressure of 50 MPa, treatment temperature of 300 °C, heating rate of 5 °C/min, processing time of 24 h, and then unloading pressure after the natural cooling to room temperature.

2.2. Characterization of thin films

The optical constants and stresses of the films were characterized. Cody-Lorentz equation is used to characterize the dielectric constant of thin films. The imaginary part of the dielectric constant is expressed as follows [19]:

$$\begin{aligned}\varepsilon_2(E) &= \mathrm{G}(E)\frac{AE_0\Gamma E}{\left(E^2-E_0^2\right)^2+\Gamma^2E^2}, \mathrm{E} > E_t \\ &= \frac{E_1}{E}\exp\left[\frac{(E-E_t)}{E_u}\right], E \le E_t\end{aligned} \tag{1}$$

$$G(E) = \frac{\left(E-E_g\right)^2}{\left(E-E_g\right)^2+E_p^2} \tag{2}$$

$$E_1 = E_tG_t(E)L_t(E) \tag{3}$$

Where E, E_0, A, E_g and Γ are photon energy, central photon energy of the interband transitions, optical transition matrix elements, ban gap and bandwidths, respectively. E_t is the boundary energy between the Urbach tail-to-tail transition and interband transitions, E_p is the second transition energy ($E_g + E_p$), the photon absorption greater than this value is Lorentz absorption line. E_u is expressed as Urbach band width, which is an important parameter to characterization the disorder degree of material microstructure and defect density of local state. E_1 is defined so that the dielectric constant of the imaginary part of ε_i is continuous at $E = E_t$; $G(E)$ is approximated by a constant density function of state density dipole. The real part ε_1 of the dielectric constant can be obtained by K–K transformation:

$$\varepsilon_1(E) = \varepsilon_\infty + \frac{2}{\pi}\boldsymbol{P}\int_0^\infty \frac{E'\varepsilon_2(E')}{E'^2-E^2}dE' \tag{4}$$