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Improving the laser damage resistance of oxide thin films and multilayers via tailoring ion beam sputtering parameters

M.B. Cosar[a,b], A.E.S. Ozhan[a,c], G.H. Aydogdu[a,*]

[a] Aselsan Inc. Microelectronics, Guidance and Electro-Optics Division, Cankırı Yolu 7. Km, 06750 Akyurt, Ankara, Turkey
[b] Middle East Technical University, Metallurgical and Materials Engineering Department, Universiteler Mah. Dumlupınar Blv. No: 1, 06800 Cankaya, Ankara, Turkey
[c] Atılım University, Graduate School of Natural & Applied Sciences, Kızılcasar Mah., 06836 Incek, 06836 Golbası, Ankara, Turkey

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ABSTRACT

Ion beam sputtering is one of the widely used methods for manufacturing laser optical components due to its advantages such as uniformity, reproducibility, suitability for multilayer coatings and growth of dielectric materials with high packing densities. In this study, single Ta_2O_5 layers and Ta_2O_5/SiO_2 heterostructures were deposited on optical quality glass substrates by dual ion beam sputtering. We focused on the effect of deposition conditions like substrate cleaning, assistance by 12 cm diameter ion beam source and oxygen partial pressure on the laser-induced damage threshold of Ta_2O_5 single layers. Afterwards, the obtained information is employed to a sample design and produces a Ta_2O_5/SiO_2 multilayer structure demonstrating low laser-induced damage without a post treatment procedure.

1. Introduction

Since the invention of the laser at 1960s, there has been a great effort to use it in various fields. The exposure of materials to coherent laser radiation can set a limit to their service life. When the radiation level reaches to a certain level, the so-called laser-induced damage threshold (LIDT), permanent effects such as melting, cracking and plasma formation are observed [1–3].

The extent of laser induced damage (LID) on thin film materials strongly depends on parameters like laser frequency, pulse duration, beam diameter, ambient temperature and beam focusing [2]. In addition, laser performance of thin films and/or multilayers can be improved by optimizing their deposition conditions. The focus of research about the development of optical systems has shifted from bulk to the surface of optical components. The coating of single and multi-layer has the lowest LIDT due to the scratches, defects, irregularities, contaminations on the surface, discontinuous of the electric field distribution at interfaces, differential expansion, stress and problems about adhesion of the layers [2,3]. Sufficient progress has not been made about elucidating the laser damage mechanisms of optical films and improving their LIDT values, yet. This is basically due to the complex nature of the process governed by many parameters such as intrinsic characteristics of the material under consideration, deposition conditions, their surface and structural properties [2–4].

In this study, dual ion beam sputtering (IBS) method was employed for the deposition of optical thin films. It is known that IBS has significant advantages in terms of good adhesion, uniformity, directionality, controllability and repeatability of the deposition. The method can produce dense, bulk-like, amorphous oxide films with low-loss optical constants [5–7]. All these properties are needed for antireflective and dielectric highly reflective multilayer optical coatings. Moreover, materials selection and coating design are critical for the resistance to the laser damage. Since most laser-induced damage is initiated at discontinuities, one way to increase the LIDT is to design the thickness of coatings which place the electric field maxima inside the coating layers and to minimize the electric field at the interfaces [2–4,8]. This may be achieved by using materials having different refractive indexes. Usually, low refractive index materials such as SiO_2 show enhanced LID resistance compared to high refractive index materials whereas the usage of high refractive index materials as Ta_2O_5, Nb_2O_5, TiO_2, ZrO_2 and HfO_2 is inevitable besides the ones having low refractive index in order to tune the reflectivity in a certain wavelength range. In addition, the above mentioned high refractive index materials demonstrate superior thermal, chemical stability and strong adhesion to their substrates. Ta_2O_5 is almost always produced with some nonstoichiometry and the oxygen deficiency in the structure can lead to a reduction in its laser damage resistance [9]. Thus, an

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* Corresponding author. Tel.: +90 5423564692.
E-mail addresses: gkuru@aselsan.com.tr, gulgun_aydogdu@yahoo.com (G.H. Aydogdu).

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Table 1
Dual ion beam sputtering deposition parameters of Ta_2O_5 single layer films.

Description	O_2 flow rate (sccm)	Substrate pre-cleaning	12-cm grid ion source	Process temperature (°C)
Process A	30	No	Yes	70
Process B	45	No	Yes	70
Process C	15	No	Yes	70
Process D	30	Yes	Yes	70
Process E	30	No	No	70
Process F	30	No	Yes	120
Process G (B + E)	45	No	No	70
Process H (B + E + D)	45	Yes	No	70
Process I (B + D)	45	Yes	Yes	70

additional heat treatment is required to decrease the O vacancy concentration in the lattice and, thereby, improve the LIDT value [9–12]. This method cannot be effective for Ta_2O_5/SiO_2 reflection and antireflection multilayers coatings because some other uncontrollable factors such as diffusion at interfaces and variations in the stress states of the layers will play a significant role. In addition, post deposition processes can drastically change the optical constants of the thin films and the optical design. As a result, the most proper tool to improve the LIDT values of the films is obviously the deposition parameters.

The main aim of this study is to deposit Ta_2O_5 thin films and Ta_2O_5/SiO_2 multilayers by IBS and investigate the influence of deposition parameters on the laser damage resistance of these coatings.

2. Experimental details

All films were deposited using dual ion beam sputtering system, equipped with two RF inductively coupled plasma ion beam sources. The ion beam source having 16 cm diameter was used for depositions whereas the one with 12 cm diameter was reserved for target pre-cleaning and assisted deposition. Layers were prepared from high purity (i.e. 99.9%) Ta and SiO_2 targets. Sputtering was performed with a typical ion beam energy and beam current of 1250 eV and 600 mA, respectively. On the other hand, energy and current used for assistance and pre-cleaning were 550 eV and 150 mA. During the deposition process, the pressure of the vacuum chamber was $\sim 1 \times 10^{-4}$ torr, O_2 flow rate was 30 sccm and Ar was the working gas. The film samples were deposited on polished L-BK7 (laser grade) glass substrates (1 or 2 inch diameter and 2 mm thick) having surface roughness values less than 1 nm. Before deposition, the substrates were cleaned by pressurized dry nitrogen, rinsed with acetone, followed by a final wipe with ethanol. Masks were placed between the sputter ion beam and the substrate holder and rotated at 300 rpm to improve the uniformity. Film thickness, three quarter wave optical thickness (QWOT) for single layer Ta_2O_5, was controlled by optical monitoring system. Surfaces of the substrates were pre-cleaned by the 12 cm diameter ion beam source for some of the specimens to remove the organic contamination. Energy and beam current of ion source were 200 eV and 100 mA, respectively. The other possible deposition parameters like O_2 flow rates and deposition temperature (the temperature variation is difficult due to cause damaging the fiber of optical monitoring system) were listed in Table 1.

Transmittance spectra of the films were measured by a Lambda 950 spectrophotometer. The refractive indexes were obtained by V-Vase ellipsometry. Microstructure was analyzed by a Rigaku X-ray diffractometer (XRD) employing Cu Kα radiation ($\lambda = 1.54056$ Å). The surface morphology and RMS roughness were measured by a NT-MDT atomic force microscope (AFM). The chemical compositions of the films were analyzed by X-ray photoelectron spectroscopy (XPS; Thermo K-Alpha) after Ar sputtering in order to remove the contaminants on the surface. LIDT were tested at 21 °C operating temperature and 42% humidity by VLOC and Quantel test laboratories. Samples are cleaned with methanol before testing. Samples are exposed to 1064 nm laser at 20 Hz repetition rate, 20 ns pulse width, linear polarization state, TEM00 beam mode and 0.7 mm beam diameter at target plane. The test sample is irradiated at several different fluence levels with 10 sites at each level and one site on sample was tested 200 times according to ISO Standard 21254-2:2011(E). The percentage of failures at each fluence is plotted against the fluence levels. A least squares linear fit to this data is calculated to determine the zero percent failure intercept which represents the damage threshold level. This method provides the most accurate measurement of damage threshold. The damage morphologies were monitored by Nomarski microscopy in dark field mode.

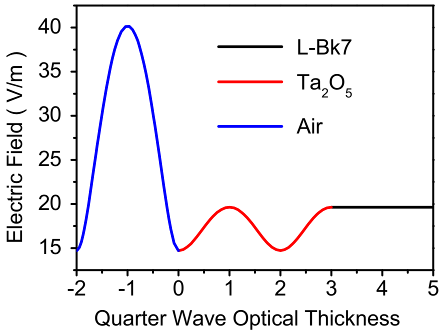

Fig. 1. Electric field distribution of three QWOT of Ta_2O_5.

3. Results and discussion

3.1. Results of Ta_2O_5 single layer films

3.1.1. Electrical field distribution analysis

Improving LIDT values starts with a successful optical design coating. Thin film design software would help to analyze electric field distribution of the coating. For single layer Ta_2O_5 film, electric field value for the medium decreases at surface of film while it increases at coating/substrate interface, shown in Fig. 1 where electric field is given V/m unit assuming the 1 W/m^2 incident power. Decreasing the electric field distribution at the interfaces leads to high-quality LIDT values, as mentioned in the literature [2,8,13]. For single layer Ta_2O_5 film, electric field value for the medium decreases at surface of film while it increases at coating/substrate interface, shown in Fig. 1. Since the interface can be sensitive to laser damage, surface of substrate should be clean and uncontaminated before deposition. It would be important in order to decrease laser damage probability.

3.1.2. Surface topography and structure analysis

Structure of all films was analyzed by XRD. As shown in Fig. 2(a), all samples deposited on glass substrate (L-BK7) were amorphous.