

Impact of LEO atomic oxygen on the optical properties and laser damage resistance of r.f. sputtered silica, niobia and tantala thin films on fused silica

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ABSTRACT

Thin films of several metal oxides commonly employed in interference coating applications were investigated with respect to their optical characteristics and laser-induced damage resistance before and after exposure to atomic oxygen under conditions representative of the low-Earth-orbit environment. High-quality thin films of Silicon dioxide, Niobium pentoxide, and Tantalum pentoxide were deposited on fused silica substrates using radio-frequency sputtering techniques. An uncoated fused silica substrate was examined concurrently for comparative purposes.

The experimental results demonstrated that atomic oxygen bombardment generally led to deterioration in both optical transmittance and laser damage resistance for all investigated samples, with the notable exception of the tantala-coated specimen. Variations in the extent of degradation or improvement among the different materials were observed. The study further discusses the interaction mechanisms between atomic oxygen and the investigated thin-film coatings, as well as the intrinsic material properties responsible for the distinct behaviors identified following exposure.

Keywords: low-Earth orbit (LEO), very-low Earth orbit (VLEO), laser-induced damage, thin films, metal oxides, optical coatings

1. INTRODUCTION

Low Earth Orbit (LEO) and very-low Earth Orbit (VLEO) represent uniquely harsh environments for optical materials, where combined exposure to atomic oxygen (AO), vacuum ultraviolet (VUV) radiation, charged particles, and thermal cycling can significantly degrade the performance and reliability of optical components used in Earth observation, laser communication, and high-energy laser systems. Among these factors, hyperthermal atomic oxygen—generated by dissociation of diatomic oxygen in the upper atmosphere at altitudes of ~200–700 km—has long been recognized as a dominant driver of surface erosion and chemical modification of spacecraft materials [1, 2, 3]. While its detrimental effects on polymers and carbon-based materials are well established [1,2], the interaction of AO with oxide thin films used in precision optics remains comparatively less explored, particularly with respect to subtle changes in optical properties and laser damage resistance.

Silica (SiO₂), niobia (Nb₂O₅) and tantala (Ta₂O₅) thin films are key constituents of high-performance optical coatings, including high-reflectivity mirrors, antireflection coatings, and filters operating from the ultraviolet to the near-infrared [4,5,6,7]. SiO₂ is widely used as a low-index, mechanically robust layer, while Nb₂O₅ and Ta₂O₅ serve as high-index materials with relatively high refractive indices, good transparency in the visible/near-IR, and compatibility with multilayer interference designs [5,6,7]. In terrestrial and laboratory environments, the optical constants, absorption losses, microstructure, and laser-induced damage threshold (LIDT) of these oxides are known to depend strongly on deposition method and conditions [7,8]. The impact of deep-space environment factors like UV, Gamma and low-energy proton radiation on the laser resistance of oxide coatings manufactured by atomic layer deposition method was investigated recently [9, 10]. Radio-frequency (r.f.) magnetron sputtering enables dense, adherent films with good thickness control,

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but can introduce intrinsic stress, substoichiometry, and defect states that are highly relevant to both optical performance and laser damage behavior [8,26,11].

For space-based laser systems—such as lidar, laser communication terminals, and high-energy laser experiments operating from LEO platforms—the long-term stability of these coatings under AO exposure is critical. Previous studies have shown that AO can induce surface roughening, mass loss, and chemical modification in a variety of inorganic and hybrid coatings [3,12,13,14]. For silica-based optics, AO and related oxidative species can alter surface morphology, modify hydroxyl content, and influence absorption centers, all of which may impact scatter and laser damage susceptibility [15,16,17]. High-index oxide coatings, including Ta₂O₅ and Nb₂O₅, are also known to exhibit structural relaxation, defect formation, and stoichiometric changes under energetic radiation and reactive species [7, **Errore. Il segnalibro non è definito.**,18], but the specific role of AO, independently and in synergy with other LEO factors, remains insufficiently characterized.

Laser-induced damage in dielectric coatings typically initiates at localized defects, compositional inhomogeneities, or nanoscale absorbers, and is strongly influenced by film density, porosity, stress, and interfacial quality [8,11,19]. Any AO-induced evolution of surface or near-surface chemistry and morphology could therefore alter both the linear optical properties (refractive index, extinction coefficient, scatter) and the nonlinear response under high-fluence laser irradiation. AO-driven formation or annihilation of defect centers, changes in stoichiometry (e.g., oxygen-rich or oxygen-deficient regions), and modification of film packing density may affect absorption at the laser wavelength and the statistical distribution of damage precursors [18,19,20]. Despite its importance for space-qualified laser optics, systematic data on how AO exposure in realistic LEO-like conditions affects the laser damage resistance of r.f. sputtered SiO₂, Nb₂O₅, and Ta₂O₅ coatings on fused silica substrates are sparse.

The present work addresses this gap by investigating the impact of simulated LEO atomic oxygen exposure on the optical properties and laser damage resistance of r.f. sputtered silica, niobia, and tantalum thin films deposited on fused silica. We examine changes in refractive index, extinction coefficient, and spectral transmission, well as surface morphology, before and after controlled AO fluences representative of LEO mission profiles. Laser-induced damage thresholds are then measured under well-defined conditions using standardized protocols [19, 21], allowing correlation of AO-induced modifications with changes in damage performance.

2. EXPERIMENTAL DETAILS

2.1 Film manufacturing

The layers were deposited using a magnetron-sputtering system (Helios, Leybold Optics), equipped with two proprietary TwinMags magnetrons and a plasma source, enabling plasma/ion-assisted reactive mid-frequency dual-magnetron sputtering. The magnetrons were optimized for both high sputtering rates and high optical-layer performance. Prior to each deposition, the chamber was evacuated by turbo-molecular pumps to a base pressure of 1×10^{-6} mbar. Silicon (Si) and tantalum (Ta) targets, with purities of 99.999% and 99.9% respectively, were used in the magnetron cathodes, with argon as the working gas and oxygen as the reactive gas for both magnetrons; the oxygen was fed close to the targets to fully oxidize the growing films. The Si cathode was operated at a constant power of 4500 W, whereas the Ta cathode was regulated in oxygen-control (lambda-control) mode to ensure stable, reproducible film stoichiometry and optical properties. The working pressure during sputtering was 1×10^{-3} mbar, and the target-to-substrate distance was 100 mm. By adjusting the electrical power applied to a cathode, the sputtering rate could be increased or decreased; we found that film quality degraded at high rates, and good optical quality was obtained at a rate of about 0.5 nm/s for both materials.

2.2 Space simulator chamber and treatments description

The samples were tested inside the space simulation chamber located at the ENEA Research Center in Brindisi (SSPT-TIMAS-MCC) [22]. The same samples were compared against the control material, Kapton, which served as the reference standard. The facility is designed to reproduce the key environmental stressors encountered in VLEO. The chamber operates with a controlled oxygen flow supplied by a dedicated high-purity O₂ line, while a cryogenic pump removes residual water vapor and light contaminants, ensuring a dry and chemically stable atmosphere throughout the tests. Thermal cycling was applied to all specimens, with temperatures oscillating between -50 °C and $+100$ °C according to a 90-minute cycle, representative of the thermal fluctuations experienced during orbital day–night transitions. The temperature of the samples was continuously monitored and recorded using Pt100 temperature probes placed on the sample holder. The

chamber is equipped with a quadrupole mass spectrometer that continuously monitors the gas composition; the detection of species such as CO, CO₂, and characteristic carbon peaks provides real-time insight into sample degradation, outgassing dynamics, and oxidation processes.

Oxygen exposure was performed in the ground-based hyperthermal beam facility in accordance with ASTM E2089 [23]. The system includes a thermocryostat-controlled sample holder equipped with a Faraday cup for current measurements and an ion gun based on a Mark I gridless ion source, capable of generating a directed oxygen flux at energies representative of orbital velocities. The samples were subjected to an oxygen ion flux of approximately 1×10^{14} ions/cm²/s, corresponding to a measured current of 40 μ A, over a total duration of 5 cycles (450 minutes). Each cycle lasted 90 minutes, consisting of a 60-minute ramp-up to +100 °C followed by a 30-minute cooldown to –50 °C. During the entire process, the accumulated ion dose reached 1.18×10^{18} ions/cm². It is important to note that the energy of atomic oxygen in the simulator the oxygen energy is approximately 70 eV.

The test chamber reproduces VLEO-relevant conditions enabling a highly realistic simulation of the oxidative and thermal stresses encountered by spacecraft materials in orbit. [24]

2.3 Characterization techniques

The direct transmittance of the samples was measured before and after the AO exposure using a Perkin Elmer Lambda 950 spectrophotometer, in the spectral range 200-2500 nm.

Surface morphology was characterized in terms of eroded material thickness and surface roughness, using a Bruker DektakXT profilometer.

The structural properties of the films were investigated by X-ray diffractometry (XRD) using a SmartLab (Rigaku) diffractometer equipped with 9 kW rotating Cu anode.

The pulsed Nd:YAG laser (model Ekspla NL 120, [25]) was used for LIDT measurements. LIDT 1-on-1 measurement was performed at 355 nm and at 1064 nm, in ns regime.

3. RESULTS AND DISCUSSION

The eroded thicknesses and the sample roughness before and after exposure to AO in the test chamber are provided in Table 1. Silicon oxide, both bulky substrate and deposited film, has shown almost 10 times higher erosion rate compared to niobia and tantalum films. Conversely, the surface roughness of both the bare substrate and the silica film increased upon the AO exposure. Instead, the AO exposure brought a certain surface smoothing to the tantalum and niobia films.

Table 1. The eroded thickness and the sample roughness before and after exposure to AO.

Sample	Eroded thickness	Roughness Ra	
		Before AO exposure	After AO exposure
	nm	nm	nm
SiO ₂ substrate	33 ± 3	0,265	0,274
SiO ₂ film	37 ± 3	0,404	1,504
Nb ₂ O ₅ film	4 ± 1	0,152	0,147
Ta ₂ O ₅ film	4 ± 1	0,236	0, 187

Figure 1(a) compares the direct transmittance of the bare fused silica substrate before and after the exposure of this sample to AO. After the treatment, the sample became more transparent, particularly in the UV range. Similar behavior demonstrated the samples with silica film and that with tantala film (not shown in the figure). The sample with niobia film showed the opposite result, as its transmittance decreased over a wide spectral range, Fig. 1(b).

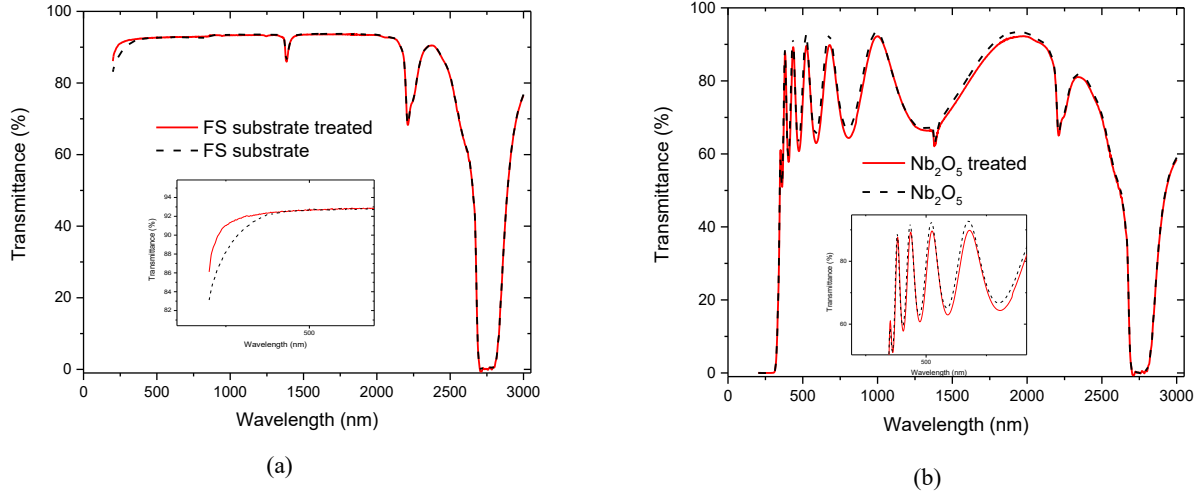


Figure 1. The optical transmittance of (a) Fused silica (FS) substrate and (b) Nb_2O_5 film on FS substrate before (dashed black line) and after (solid red line) the AO exposure.

The r.f.-sputtered microstructure—governed by parameters such as substrate temperature, ion bombardment, and oxygen partial pressure—sets the initial porosity, stress, and defect population that mediate AO uptake and laser coupling. XRD investigation proved polycrystalline microstructure for our silica and tantala films, while our niobia film was amorphous. When atomic oxygen interacts with a metal oxide surface, oxygen vacancies can be filled, which enhances film stoichiometry and optical transparency while reducing absorption losses—especially when films are deposited or treated under oxygen-rich or ion-assisted conditions. This should be true also for Nb_2O_5 [26, 27], however the opposite was observed for our niobia film. This may be explained by the formation of sub-stoichiometric phases such as NbO_2 or NbO , caused by excessive oxygen incorporation previously observed for certain temperature and pressure conditions [28]. Niobium pentoxide thin films may exhibit a significant capacity to accommodate atomic oxygen, influenced by their amorphous or polymorphic crystal structures. The capacity for oxygen incorporation stems from Nb's variable oxidation states (ranging from +2 to +5), enabling oxygen intercalation, defect formation, and slight non-stoichiometry ($\text{Nb}_2\text{O}_{5-\gamma}$). This flexibility allows the films to sustain oxygen-rich environments without rapid phase degradation.

The results of LIDT tests at 355 nm laser wavelength in ns regime are summarized in Table 2. For all samples except for the tantala film, the AO exposure brought decrease in laser damage resistance.

Table 2. The results of laser damage tests at 355 nm.

Sample	LIDT J/cm ²
Tantala film	0.11
Tantala film treated with AO	2.68
Silica film	9
Silica film treated with AO	4.11
Niobia film	0.05
Niobia film treated with AO	0.014
FS substrate	19.82
FS substrate treated with AO	2.43

As-deposited tantalum and niobium showed similar LIDT values, but their exposure to AO resulted in opposite effects. Tantalum increased its LIDT by almost 25 times, while niobium resistance decreased by 5 times. Modification of the oxidation states and the surface relaxation are the key factors underpinning this phenomenon.

For the FS substrate and the silica film of FS, the LIDT decrease is mainly due to surface erosion and possible formation of oxygen vacancies due to erosion. Interestingly, the initially much higher LIDT of FS substrate dropped down dramatically upon the AO exposure, by 10 times, while the silica film lost only half of its resistance, so that the coated sample of the same material overperforms the bare substrate.

4. CONCLUSIONS

This work investigates how controlled AO exposure characteristic of LEO and VLEO alters the optical properties, surface morphology, and laser damage resistance of r.f.-sputtered SiO₂, Nb₂O₅, and Ta₂O₅ films deposited on fused silica. By correlating spectrophotometry for optical response, profilometry for precise surface mapping, and standardized LIDT testing before and after AO exposure, we infer the structure–property relationship that governs in-orbit optical stability.

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