

天基激光雷达用 532 nm/1064 nm 双波段反射膜研制

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摘 要: 天基激光雷达技术是目前测绘、国防等领域重要的侦测手段。532 nm/1 064 nm 双波段反射膜是激光雷达中的重要元件, 负责光路的传输和光谱效率的调控。目前, 电子束蒸发工艺 (Electron Beam evaporation, EB) 是制备高损伤阈值 $\text{HfO}_2/\text{SiO}_2$ 双波段反射膜的主要技术手段。但是, 受制备工艺沉积能较小的限制, $\text{HfO}_2/\text{SiO}_2$ 薄膜疏松多孔, 在大气环境下容易吸附水汽, 这些水汽会在真空工作环境下解吸从而导致薄膜产生谱移, 影响激光雷达的工作稳定性。为了降低薄膜元件在大气和真空环境下由于水汽造成的影响, 文中利用原子层沉积技术 (Atomic Layer Deposition, ALD) 在 EB- $\text{HfO}_2/\text{SiO}_2$ 多层膜的顶部和侧壁覆盖了一层 20 nm 的 Al_2O_3 薄膜, 由于原子层沉积自下而上的自然生长机制决定了薄膜的致密无针孔特性, 同时具有优异的保形性、均匀性、厚度可控性等, 不仅可以阻隔水汽的渗透, 且能够保证薄膜具有较高的损伤阈值。通过测试结果表明, EB- $\text{HfO}_2/\text{SiO}_2$ 覆盖 ALD- Al_2O_3 后在真空-大气环境下的谱移仅有 0.3%, 低于覆盖前的 2.5%, 同时在 532 nm 和 1064 nm 处的抗激光损伤阈值分别为 13.1 J/cm^2 和 41.5 J/cm^2 , 可以满足激光雷达的应用要求。

关键词: 激光雷达; 双波段反射膜; 电子束蒸发; 原子层沉积; 损伤阈值

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0 引 言

天基激光雷达技术在水下目标识别、海洋测绘及战场环境评估等领域扮演着不可替代的重要作用^[1]。双频激光雷达广泛应用于天基探测领域, 能够同时输出 1064 nm 的基频纳秒脉冲激光和 532 nm 的倍频纳秒脉冲激光, 从而有效实现对地表、海表和海底回波的探测^[2-3]。其中, 1064 nm/532 nm 双波段高反射薄膜元件在双频激光雷达中起着关键作用, 负责光路的传输和光谱效率的调控^[4-5]。为了提升雷达探测目标的精度、范围和响应速度, 探测激光需要具备高的能量和重频。因此, 双波段反射膜元件必须具备高损伤阈值, 以确保整个系统的稳定性^[6-7]。另一方面, 不同型号的天基激光雷达位于不同的轨道高度, 其服役真空度不同。因此, 双波段反射膜元件还需要能够在不同的真空环境下保证稳定的光谱效率^[8]。

电子束蒸发 (Electron Beam evaporation, EB) 是在真空条件下通过电子束直接加热膜料使之气化并在基底上成膜的技术。采用电子束蒸发工艺沉积 $\text{HfO}_2/\text{SiO}_2$ 多层膜被广泛应用于纳秒脉冲强激光薄膜的制备。已经证实, 通过这种工艺制备的薄膜器件具有最高的抗激光损伤阈值^[9-10]。但是, EB 工艺存在粒子沉积能量较低的问题, 导致薄膜结构疏松多孔。当镀膜设备开门进气以后, 薄膜会迅速吸收空气中的水汽, 从而填满膜内的孔隙, 这个过程会导致薄膜的折射率和光学厚度增加; 而当薄膜从大气环境转移到高真空环境后, 薄膜孔隙中的水汽会排出, 这一过程中, 薄膜的折射率和光学厚度会降低。真空度不同, 水汽析出程度不同, 薄膜的光学特性不同。这意味着, 即使 EB- $\text{HfO}_2/\text{SiO}_2$ 有高的损伤阈值, 但在天基激光雷达中广泛应用却具有困难。因此, 如何提升 EB- $\text{HfO}_2/\text{SiO}_2$ 的真空稳定性, 是制备高性能天基激光雷达用 1064 nm/

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532 nm 双波段高反射薄膜元件的关键问题。

提高薄膜的防水汽性能和真空稳定性目前已经有一些研究报道。JENSEN L 等人采用电子束蒸发、离子辅助沉积 (Ion Assisted Deposition, IAD) 和离子束溅射 (Ion Beam Sputtering, IBS) 制备了多层增透膜^[11], 在真空条件下。发现只有 EB 工艺制备的薄膜存在显著的波长偏移和降低的损伤阈值, 展现了 IAD 和 IBS 工艺制备防水汽性能优良和真空稳定薄膜的潜力^[12]。然而, IAD 工艺虽然可以提高薄膜的堆积密度, 高能沉积容易使 Hf-O 键断裂, 引起较大的膜层缺陷吸收, 影响薄膜损伤阈值。而 IBS 工艺制备成本高、成膜速率慢、薄膜应力大, 且难以制备大口径薄膜元件。以上短板严重限制了这两种工艺在天基激光雷达中的应用。OLIVER 等人在 $\text{HfO}_2/\text{SiO}_2$ 多层膜中加入了 Al_2O_3 补偿层, 能够降低水的渗透, 然而这种方法具有较强的时间依赖性, 难以保证长期稳定^[13]。CHENG 等人^[14]通过双源混合共蒸发的方式制备了 $\text{Hf}_x\text{Si}_{1-x}\text{O}_2$ 混合膜, 可以兼顾高损伤阈值和致密防水性能, 但共蒸发工艺复杂、成本高, 难以精准控制。ZENG^[15-16]等人在 EB- $\text{HfO}_2/\text{SiO}_2$ 基础上采用 PIAD(等离子体离子辅助沉积) 在顶部和侧壁制备了 SiO_2 层, 获得了光谱长期稳定的薄膜, 但其顶部膜层应力不匹配, 容易膜裂从而降低防护效果。综上所述, 目前尚缺少一种高损伤阈值、良好真空稳定性的天基激光雷达用双波段高反射薄膜元件的制备方法。

原子层沉积 (Atomic Layer Deposition, ALD) 是基于自限制界面反应的薄膜生长技术, 可以制备结构致密、高保形、低缺陷密度、均匀性好的薄膜^[17-18]。氧化铝是原子层沉积最常见的薄膜 (ALD- Al_2O_3), 具有高透明度、高禁带宽度、非晶态、高阻隔性以及良好的化学和热稳定性, 目前已广泛用于高阻隔柔性电子器件的封装^[19]。GRONER 等^[20]在聚酰亚胺基材上利用 10~25 nm 的 ALD- Al_2O_3 薄膜将水蒸气透过率 (WVTR) 降低至 $1 \times 10^{-3} \text{ g/m}^2 \cdot \text{day}$ 。GEROGY 等^[21]利用 T-ALD 在柔性有机聚酯 (PEN) 基底上低温沉积 20 nm Al_2O_3 , 其水蒸气的透过率低于 $10^{-4} \text{ g/m}^2 \cdot \text{day}$ 。然而, 目前 ALD- Al_2O_3 在激光薄膜领域的封装还鲜有报道。

文中提出了一种新的防水汽激光薄膜的制备方法。首先采用电子束蒸发制备 $\text{HfO}_2/\text{SiO}_2$ 多层膜, 之后利用原子层沉积技术在多层膜顶部和侧壁覆盖了 20 nm 的 Al_2O_3 作为水汽阻隔层, 测试并表征了覆盖

前后薄膜的光谱漂移和损伤阈值的变化, 实验结果为天基激光雷达中的大气-真空稳定薄膜的研制提供了有效参考。

1 实验

1.1 薄膜设计

主膜系选择电子束蒸发制备的 532 nm 和 1064 nm 双波段 0° 高反射薄膜。膜层基础结构为 $\text{sub} | (0.65\text{H}1.35\text{L})^{20} | \text{A}$, 其中 H 代表光学厚度为 $1/4\lambda$ 的 HfO_2 薄膜, L 代表光学厚度为 $1/4\lambda$ 的 SiO_2 薄膜。在优化光谱时, 考虑到在用电子束蒸发制备 $\text{HfO}_2/\text{SiO}_2$ 多层膜后由于其柱状多孔的结构已经吸附了一定的水汽, 随后在原子层沉积制备过程中由于抽真空产生水解吸, 因此此时的光谱会向短波方向漂移, 这就导致真实光谱与设计时的光谱有偏差。经过多次实验测试后, 在设计时通过调整参考波长将主膜系的中心波长从 1064 nm/532 nm 向长波方向偏移了 3%, 从而修正了水解吸引入的光谱偏差。

之后对薄膜的电场进行调控。由于界面处缺陷相对密集, 机械稳定性较差^[22], 因此纳秒激光的损伤阈值受界面影响很大, 所以需要将界面调整在薄膜内部电场强度较低的位置^[23-24]。在 $\text{sub} | (0.65\text{H}1.35\text{L})^{20} | \text{A}$ 结构的基础上, 增加 0.5L 改变最外层低折射率材料的光学厚度, 可以有效降低膜层的界面电场强度。最终设计得到的膜层结构为: $\text{sub} | (0.65\text{H}1.35\text{L})^{20} 0.5\text{L} | \text{A}$ 。考虑到真空环境下水解吸引起薄膜折射率的降低, 文中将材料的折射率相应降低 3% 以对应真空环境。图 1 和图 2 分别展示了 0° 入射时的对比设计光谱以

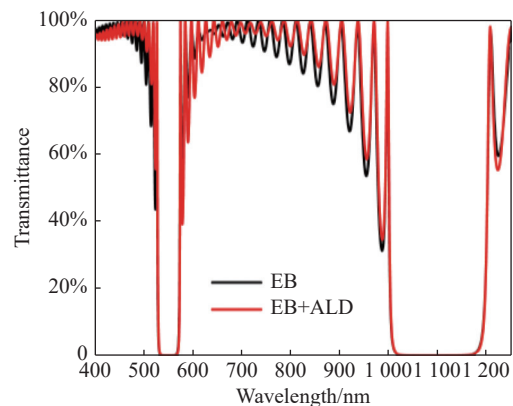


图 1 在 0° 入射时的设计光谱

Fig.1 Design spectrum at 0° incidence

及电场分布,可以看出在覆盖 ALD- Al_2O_3 前后差异很小。

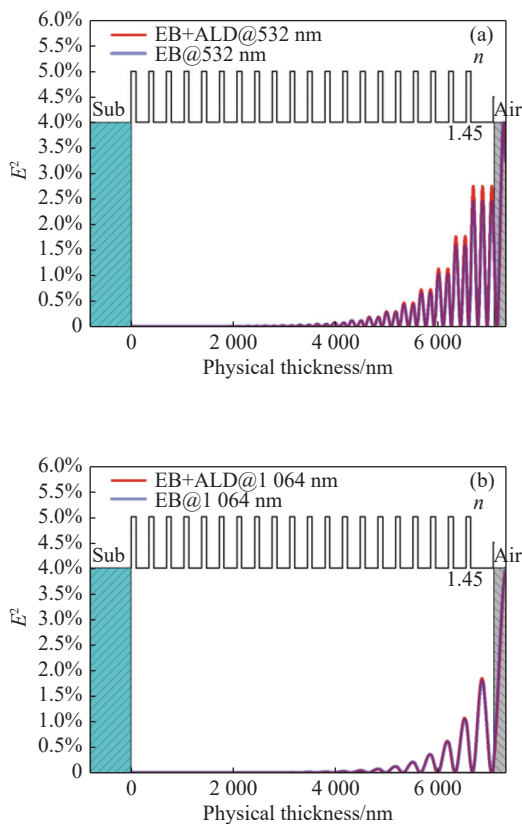


图2 在 0° 入射时 (a) 532 nm 和 (b) 1064 nm 激光辐照下的电场分布模拟

Fig.2 Simulation of electric field distribution under laser irradiation at (a) 532 nm and (b) 1064 nm at 0° incidence

1.2 薄膜样品制备

主膜系采用光驰公司生产的 OTFC-1300 型镀膜机制备。在薄膜制备时,沉积温度为 150°C ,本底真空优于 $4.0\times 10^{-4}\text{ Pa}$,恒温时间为 120 min。所有膜层均采用电子束蒸发工艺制备。在蒸镀过程中,沉积速率保持恒定, HfO_2 的蒸发速率均为 0.2 nm/s , SiO_2 的蒸发速率均为 0.8 nm/s 。

随后,利用原子层沉积技术 (NCR-200 R 体系, nanofrontier, China) 在反射主膜系的膜层表面、膜层侧壁沉积一层厚度为 20 nm 的 Al_2O_3 薄膜,前驱体采用 O_3 -TMA 标准双脉冲体系,一个循环内控制为 0.1 s O_3 脉冲,5 s 冲扫和 0.1 s TMA 脉冲,2 s 冲扫,沉积速率约为 0.08 nm/cycle 。控制基板温度为 80°C 。膜层设计如图 3 所示。

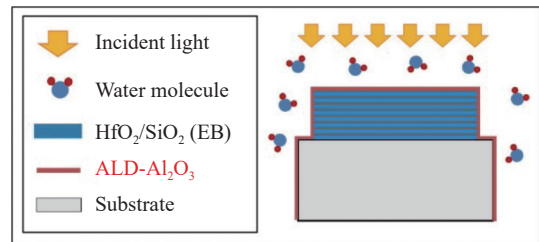


图3 膜层示意图

Fig.3 Schematic diagram of the film layer

1.3 样品表征

用 CARY5000 型分光光度计测量薄膜在大气环境下的透射率光谱。利用美国 Veeco 公司生产的 Spector 双离子束溅射镀膜机腔体内部光谱仪测试薄膜在真空环境下的透射率光谱,确定光谱漂移量。测试波长为 450~1300 nm。

采用自搭建的损伤阈值测试平台对薄膜的损伤阈值进行测试。其中,所采用激光器为 Spectra Physics 公司的 Nd: YAG 激光器。输出波长为 1064 nm 时,5000 mm 的聚焦透镜焦点处,高斯光峰值 $1/e^2$ 处光斑的半径为 $987.6\text{ }\mu\text{m}$,90% 峰值处半径 $155.4\text{ }\mu\text{m}$,最大输出能量约 700 mJ,调 Q 脉冲宽度为 10 ns。输出波长为 532 nm 时,5000 mm 的聚焦透镜焦点处,高斯光峰值 $1/e^2$ 处光斑的半径为 $752.9\text{ }\mu\text{m}$,90% 峰值处半径 $119.5\text{ }\mu\text{m}$,最大输出能量约 500 mJ,调 Q 脉冲宽度为 8.5 ns。两种波长激光重频均为 10 Hz。测试时样品均放置于真空罐中,保持真空度优于 $3\times 10^{-4}\text{ Pa}$ 。

2 结 果

2.1 透射光谱

在大气环境中,电子束蒸发制备的薄膜疏松多孔,吸附水汽导致整体折射率变大、光学厚度增加从而使得光谱向长波方向移动。此外,由于薄膜的微观状态与制备工艺密切相关,不同工艺或设备下薄膜的孔隙率存在差异,同时环境条件也会影响薄膜的吸潮量。因此,当薄膜在真空工作环境发生水解吸后这种光谱的不稳定可能导致激光雷达的效率降低甚至失效。为获得大气到真空的稳定光谱,文中在 EB 多层膜上沉积一层致密的 ALD- Al_2O_3 从而降低水汽对薄膜的渗透。通过光谱仪测试了 EB 制备的 $\text{HfO}_2/\text{SiO}_2$ 多层膜和覆盖 20 nm 的 ALD- Al_2O_3 后在大气中放置一周后的透射光谱,在 $3\times 10^{-4}\text{ Torr}$ ($1\text{ Torr}=133.322\text{ Pa}$)

的真空室内测量了样品的真空光谱。结果如图 4 和表 1 所示。

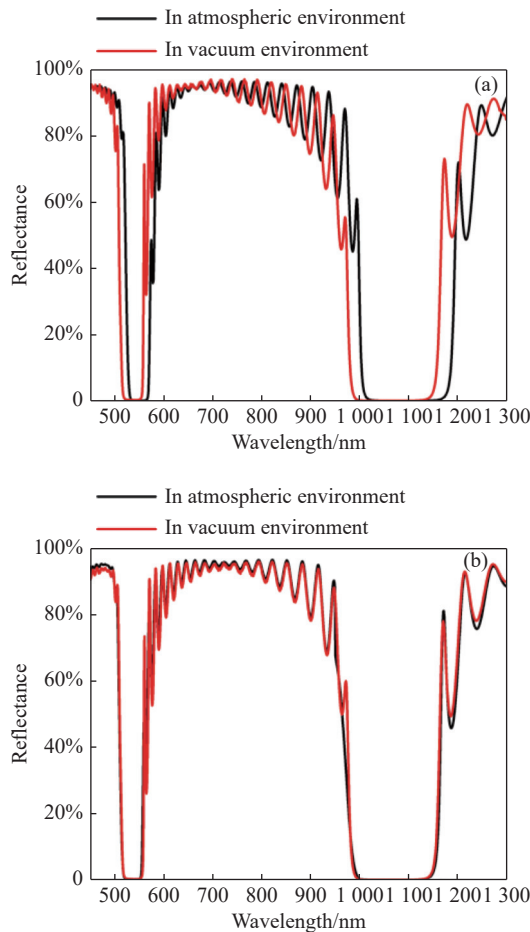


图 4 真空和大气环境中的光谱。(a) 未覆盖 ALD- Al_2O_3 的 EB 多层膜；(b) 覆盖 ALD- Al_2O_3 后的多层膜

Fig.4 Spectra in vacuum and atmosphere. (a) EB multilayer film before ALD- Al_2O_3 coverage; (b) EB multilayer film after ALD- Al_2O_3 coverage

表 1 真空和大气中的光谱漂移量

Tab.1 Spectral drift in vacuum and atmosphere		
Preparation method	Spectral shift	Average shift ratio
EB	28 nm@1064 nm	2.5%
	13 nm@532 nm	
EB+ALD	3 nm@1064 nm	0.3%
	1 nm@532 nm	

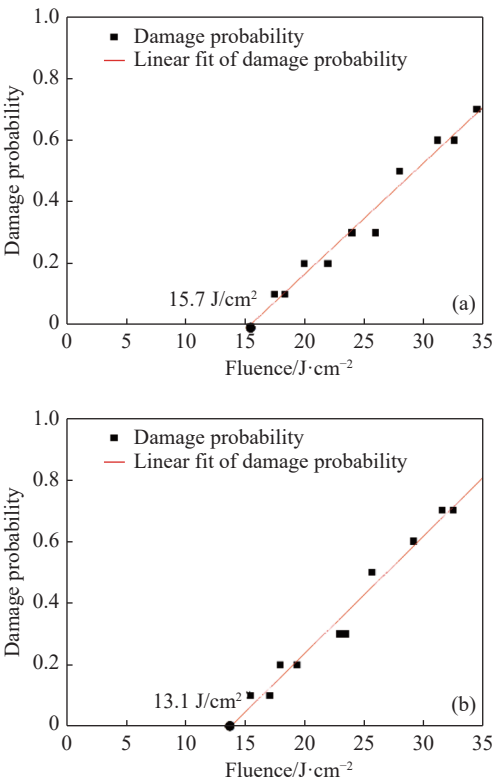
从结果中可以看出, EB 工艺制备的薄膜由于疏松多孔的结构会在大气环境中吸附大量水汽, 而在真空环境下水解吸使得孔隙中 $n=1.33$ 的水替换为 $n=1$ 的空气, 导致薄膜光学厚度减少, 光谱向短波方向漂

移, 在中心波长 1064 nm 和 532 nm 处产生了约为 2.5% 的整体漂移。而在顶部和侧壁覆盖 20 nmALD- Al_2O_3 后, 由于致密的氧化铝层基本没有开放孔隙, 可以避免水汽对薄膜的渗透, 因此只产生了极小的漂移。此外, 不同样品在大气与真空环境下的带宽保持稳定, 同时在真空工作环境中覆盖 ALD 膜后反射效率并未下降。光谱结果表明覆盖 20 nmALD- Al_2O_3 层后的薄膜在真空-大气环境下具备较强的光谱稳定性。

2.2 激光损伤阈值

采用 10-on-1 的测试方式来评价薄膜的损伤阈值, 测试的点与点间隔 1 mm。随着能量的增加, 损伤几率也在增加, 在出现损伤的镜片上只进行一种能量测试, 取零损伤几率时的能量密度作为样品的损伤阈值。测试时样品位于真空罐中, 测试结果如图 5 和表 2 所示。

从结果中观察到, 532 nm 激光辐照下, 覆盖 ALD- Al_2O_3 后的多层膜的损伤阈值 (13.1 J/cm^2) 要略低于覆盖前的损伤阈值 (15.7 J/cm^2)。1064 nm 激光辐照下, 覆盖 ALD- Al_2O_3 后的多层膜的损伤阈值 (41.5 J/cm^2) 也略低于覆盖前的损伤阈值 (44.5 J/cm^2), 但整体的阈值水平依然相近。损伤阈值降低的原因一方面可能



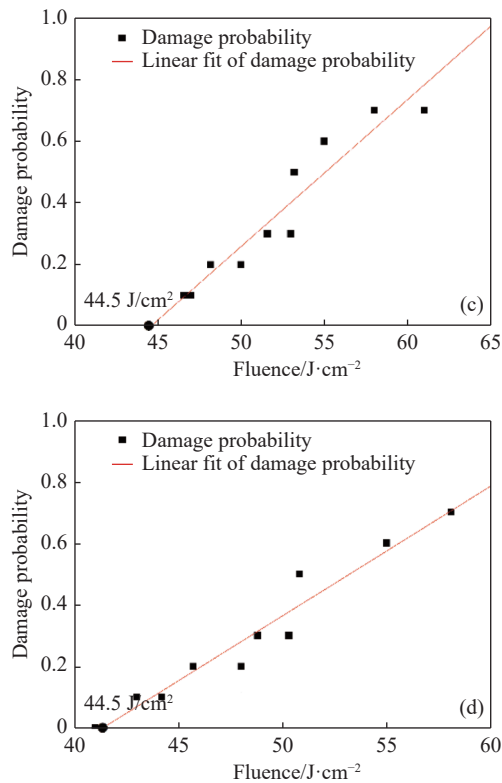


图5 真空下 532 nm 激光下的损伤阈值: (a) 未覆盖 ALD-Al₂O₃ 的 EB 多层膜; (b) 覆盖 ALD-Al₂O₃ 后的多层膜。以及 1064 nm 激光下的损伤阈值: (c) 未覆盖 ALD-Al₂O₃ 的 EB 多层膜; (d) 覆盖 ALD-Al₂O₃ 后的 EB 多层膜

Fig.5 Damage threshold at 532 nm laser: (a) EB multilayer film before ALD-Al₂O₃ coverage; (b) EB multilayer film after ALD-Al₂O₃ coverage. And damage threshold at 1 064 nm laser: (c) EB multilayer film before ALD-Al₂O₃ coverage; (d) EB multilayer film after ALD-Al₂O₃ coverage

表 2 真空环境中的损伤阈值

Tab.2 Damage threshold in vacuum	
Preparation method	Laser damage threshold
EB	44.5 J/cm ² @1064 nm
	15.7 J/cm ² @532 nm
EB+ALD	41.5 J/cm ² @1064 nm
	13.1 J/cm ² @532 nm

是 ALD-Al₂O₃ 自身吸收较大,笔者等将在后续的工作中,对 ALD-Al₂O₃ 单层膜的薄膜特性进行进一步的表征和研究;另一方面,采用 EB 和 ALD 两种制备工艺相结合的技术方案,在二次镀膜时,薄膜元件运转、清洗、上架等流程中不可避免会引起灰尘等杂质的吸附,使得外层 ALD-Al₂O₃ 与 EB-SiO₂ 膜层界面处存在

较多缺陷,导致局部电场增强,影响薄膜损伤阈值。整体来看,覆盖 ALD-Al₂O₃ 后相较于覆盖前在 1064 nm 及 532 nm 处损伤阈值变化的程度较小,薄膜的损伤阈值依然能够达到实际工作要求。

3 结 论

文中结合了电子束蒸发和原子层沉积两种制备方法,在电子束蒸发制备的 HfO₂/SiO₂ 多层膜基础上,在顶部和侧壁用原子层沉积制备厚度为 20 nm 的 Al₂O₃ 薄膜作为水汽阻隔层。结合对光谱、损伤阈值的表征,数据结果说明薄膜表面的 ALD-Al₂O₃ 层能有效阻隔水汽的渗透,在大气和真空环境中基本没有光谱漂移。同时,薄膜整体的激光损伤阈值较高,说明 20 nmALD-Al₂O₃ 层的引入对整体阈值影响较小。文中的方法和结果为提升天基激光雷达中薄膜的在真空-大气环境下的稳定性提供了重要参考。以后的工作中,将来会进一步攻关清洗工艺,将亚微米级的缺陷控制住,期望获得更高的损伤阈值。

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Research of 532 nm/1 064 nm dual-band high reflective coatings applied in space-based lidar

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Abstract:

Objective Space-based laser radar (lidar) technology is widely acknowledged as an indispensable detection method in critical fields such as geospatial mapping and national defense. The 532 nm/1064 nm dual-band reflectance coating constitutes a vital component within lidar systems, performing essential functions in optical path transmission and the regulation of spectral efficiency. Electron beam evaporation (EB) is the predominant technique employed for the fabrication of HfO₂/SiO₂ dual-band reflective coatings, which are characterized by high damage thresholds. However, due to the inherent limitations of the fabrication process, HfO₂/SiO₂ thin films

typically exhibit a porous and loosely compacted microstructure, rendering them highly susceptible to the adsorption of water vapor under atmospheric conditions. The subsequent desorption of this water vapor under vacuum operating environments leads to spectral shifts within the thin films, which significantly compromises the operational stability and performance reliability of the laser radar system. Therefore, it is necessary to improve the current preparation method to meet the needs of different usage environments. For this purpose, a waterproof vapor laser film is designed in this paper.

Methods The $\text{HfO}_2/\text{SiO}_2$ multilayer film is designed and fabricated using electron beam evaporation, followed by the deposition of a 20 nm Al_2O_3 layer on the top and side walls of the multilayer as a water vapor barrier, using atomic layer deposition (ALD) (Fig.3). The performance of the water vapor barrier is evaluated by testing the spectral characteristics in both vacuum and atmospheric conditions (Fig.4). The laser damage threshold at 1 064 nm and 532 nm is assessed using a Nd:YAG laser to evaluate the laser performance (Fig.5).

Results and Discussions Upon testing the thin films under both vacuum and atmospheric conditions, it is observed that the films fabricated via the electron beam (EB) process exhibit an average overall drift of approximately 2.5% at the central wavelengths of 1 064 nm and 532 nm (Fig.4(a)). In contrast, when the top and side walls are coated with a 20 nm layer of ALD- Al_2O_3 , only a negligible drift of 0.3% is observed (Fig.4(b)). This demonstrates excellent vapor barrier performance. Moreover, the bandwidth of the samples remains stable in both atmospheric and vacuum conditions, with no discernible reduction in reflection efficiency following the deposition of the ALD- Al_2O_3 layer in the vacuum environment. Furthermore, the damage threshold of the multilayer film with the ALD- Al_2O_3 coating (13.1 J/cm²) is found to be slightly lower than that of the multilayer film without the ALD- Al_2O_3 coating under 532 nm laser irradiation (15.7 J/cm²) (Fig.5(a)-5(b)). Under 1 064 nm laser irradiation, the damage threshold of the multilayer film after ALD- Al_2O_3 coverage (41.5 J/cm²) is observed to be marginally lower than that of the uncoated film (44.5 J/cm²) (Fig.5(c)-5(d)). The current results are sufficient to fulfill the practical operational requirements.

Conclusions A waterproof vapor laser film has been prepared. Due to the dense microstructure of the Al_2O_3 film prepared by atomic layer deposition, it was covered on the top and sidewalls of the $\text{HfO}_2/\text{SiO}_2$ multilayer film prepared by electron beam evaporation. Testing the spectrum under atmospheric-vacuum conditions showed that the drift caused by water vapor decreased from 2.5% to 0.3%, demonstrating good water vapor barrier performance. Additionally, the laser damage threshold test of the film showed that after covering with the ALD film, the threshold decreased from 15.7 J/cm² at 532 nm to 13.1 J/cm², and from 44.5 J/cm² at 1 064 nm to 41.5 J/cm². This may be due to defect attachment during the transport process. Nevertheless, this waterproof vapor laser film still meets operational requirements and enhances atmospheric-vacuum stability.

Key words: lidar; dual-band high reflective coating; electron beam evaporation; atomic layer deposition; damage threshold