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## Marine environment compatible antireflection coating with nanotop layer on silicon optics



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• Antireflection (AR) coating on silicon optic has critical requirement in thermal devices.

• The design for AR coating was finalized with optimized layers and thicknesses.

• Mechanical strength of the coating is improved by a nanotop layer.

• Salt spray and salt solubility test were conducted on sample.

#### ARTICLE INFO

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#### ABSTRACT

Antireflection coating on silicon optics have crucial importance in thermal device working in 3.6–4.9  $\mu$ m wavelength region. When the thermal device is used in marine environment, the optics face harsh saline weather condition compared to normal field environment. This deteriorates coated optics and to improve mechanical strength of the coating, a nanotop layer on the antireflection coating has been developed. In this paper a study has been carried out to improve marine environment compatibility by employing a nanolayer on the top of antireflection coating on silicon optics. Optimac synthesis method was used to design the multilayer stack on the substrate with germanium and IR-F625 as high/low refractive index respectively and the layer number was restricted to four layers. The top nanolayer was  $60 \pm 2$  nm thick hafnium dioxide layer developed with ion assisted deposition (End–Hall) on the optics during coating process. The deposition of multilayer coating was carried out at high vacuum ( $2-6 \times 10^{-6}$  mbar)) using electron beam gun and layer thicknesses were measured with crystal monitor. The average transmission achieved was 97% in the spectral band of 3.6–4.9  $\mu$ m with a hardness of 9.7 GPa on the coated optics.

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#### 1. Introduction

Most targets of civilian interest, defence interest, vehicles, airfields, factories etc. differ from the general terrain either in temperature or emissivity or both. They have different radiating characteristics which are difficult to camouflage and which can easily be seen by thermal systems. In order to detect hot objects, the 3–5  $\mu$ m window is found to be most suitable and it is one of the important atmospheric transmission window used for thermal imaging [1,2]. In Mid-wave infrared (MWIR) spectral region, silicon and germanium optics are mostly used. Silicon's high refractive index, low dispersion and easy fabrication make it very useful optical component. On the other hand, its surface hardness, robust mechanical strength along with non-hydroscopic and non-toxic properties makes it a strong candidate for optical element in 3–5 µm region. Some works were reported in the journals for silicon antireflection coating [3–6]. MWIR is used for target identification under all typical environmental conditions including fog, artic, tropical, desert, oil fog sandstorm and maritime [7]. In recent years, the demand for electro-optical system operating in the infrared region of the electromagnetic spectrum is on the increase, which has further gone up for optical systems having high transmission in marine environment conditions. It has been found that thermal imaging system (3.6-4.9 um) used in saline weather situation. normal field conditioned optic gets deteriorated due to salty environment. It is observed when the deposited coating material is having nanoorder thickness certain characteristics (like mechanical strength, colour, electric and magnetic properties) of the material are enhanced or altered which can be applied in thin film coating



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to improve coating characteristics [8]. In this paper nanolayer was used to improve mechanical strength of the coating to resist harsh marine conditions.

This paper reports design and fabrication of marine environment compatible antireflection coating  $(3-5\,\mu m)$  on silicon substrate with a top nano-protective layer. The nanolayer provides coating hardness without affecting much to the transmission value.

#### 2. Design considerations

The design of antireflection coating was based on comprehensive search method [9]. In this method the desired characteristics (transmission/reflection) were determined by optimizing the layer thicknesses of high and low refractive index layers. Optimization of design was carried out with the help of commercial computer program called Essential Macleod. Refinement and synthesis techniques are automatic process for the improvement of design performance. Refinement technique begins with an initial design and iteratively refines it to achieve a better design and synthesis technique generates a film design based on the desired film characteristic only. The software provides six distinct classes of technique for refinement and/or synthesis. One of them is Optimac, it is both a refinement and a synthesis technique and developed by Thin Film Centre Inc. Optimac is a powerful technique, it will add and remove layers in a design by successive iterations methods, modifying the thickness of successive layers slightly each time, in order to meet the required specification and improve an existing design or to generate a design from a specification. In this manner the optimized number of layers was achieved for the selected coating material combination.

Selection of coating materials was based on their refractive index values, physical stability characteristics, their transparency in the desired wavelength regions and interface compatibility. The coating materials chosen were germanium as a high index and IR-F625 as a low index. IR-F625 (transparency range 0.2-16  $\mu$ m) was procured from *M*/*S* Umicore materials having melting point 1250 °C. The best possible design for silicon antireflection coating was evaluated from lowest possible value of merit function with minimum possible number of layers, their refractive indices and corresponding optimized optical thicknesses. On the top of antireflection coating a nanolayer of hafnium dioxide  $(60 \pm 2 \text{ nm})$ was used as protective layer. The thickness of protective layer is established after several trials to obtain best possible mechanical strength. The thickness of nanolayer was very small compared to design wavelength  $(\lambda)$ . So the nanolayer thickness does not affect the transmission value of antireflection coating stack and it provides improvement in environmental durability in marine condition. The thickness verses refractive index profile is shown in Fig. 1 and theoretical transmission curve in Fig. 2 below.

#### 3. Experimental highlights

The multilayer stack designed for the antireflection coating on silicon substrate has been fabricated by using electron beam evaporation system in Symphony 9 (supplied by *M/S* Tecport Optics Inc.) vacuum coating plant. In this plant the cryo pump is used to minimize water vapor in the vacuum process and the residual gas analyzer (RGA) system of the plant shows the partial gas pressure of various gas levels present inside the coating plant. The RGA helps to identify the presence of unwanted gases during deposition. The substrates were cleaned using ultrasonic cleaning process followed by vapor degreaser cleaning. The job was rotated with planetary rotation inside the chamber for uniformity. The substrate was heated up to 200 °C (with tolerance +5 °C) for two hours inside the vacuum chamber. Particularly germanium material was coated at substrate temperature below 200 °C to avoid absorption [10].

Inside the vacuum chamber the optics were subjected to ionic cleaning for thirty minutes with the help of End-Hall ion gun. Parameters for ion cleaning are: Bias current (I<sub>B</sub>): 5A, Discharge current ( $I_{D}$ ): 4.5A, Ar flow: 12 sccm, Discharge voltage ( $V_{D}$ ): 100 V, Keeper current  $(I_k)$ : 1.6A. The evaporation took place at the working vacuum range  $6 \times 10^{-6}$ -2  $\times 10^{-6}$  mbar for germanium and IR-F625. For hafnium dioxide the evaporation took place at the working vacuum range  $4 \times 10^{-4}$ – $2x10^{-4}$  mbar in presence of oxygen gas. During the coating process of nanolayer the coated optics was exposed with oxygen ions from End-Hall ion gun system. The nanolayer was deposited at 225 ± 5 °C and nanolayer thickness with improved durability was evaluated with trial and error method. Parameters for ion assisted deposition are: Bias current (I<sub>B</sub>): 6A, Discharge current (I<sub>D</sub>): 5A, O<sub>2</sub> flow: 12 sccm, Discharge voltage ( $V_{\rm D}$ ): 120 V, Keeper current ( $I_{\rm k}$ ): 1.5A. The optimum value of nano-layer thickness is 60 ± 2 nm and hardness of the coating is 9.7 GPa + 0.31 (measured by nanoindenter supplied by *M/S* Micro Materials, UK). The rate of evaporation of the coating material is a critical parameter responsible for smooth index profile. The rate of evaporation in case of IR-F625 was 8 Å/ s and for germanium 4 Å/s and for hafnium oxide 1 Å/s. Several trials were conducted to optimize the top layer thickness for optimum value of hardness without affecting the transmission value.

#### 4. Results and discussion

The coated sample of silicon was measured for transmission with *M*/S Perkin Elmer supplied Fourier transform infrared spectrophotometer of model GX-Optics. The experimental transmission



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