



Functional multi-layer nitride coatings for high temperature solar selective applications



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ARTICLE INFO

Article history:

Received 15 February 2013

Received in revised form

28 September 2013

Accepted 22 October 2013

Available online 14 November 2013

Keywords:

Solar selective coatings

Multi-layer thin films

Cylindrical cathodic arc deposition

TiAlCrN

TiAlN

AlSiN

ABSTRACT

A solar-selective functional multi-layered coating for high temperature applications has been developed on copper and stainless steel substrates using an industrially viable cathodic arc physical vapor deposition technique. The selective coating was formulated in such a way that the constituent functional layers are mainly high temperature stable nitride materials which are also hard and capable of imparting protection against wear. A multi-layer structure comprising TiAlCrN as IR reflector, TiAlN as absorber and AlSiN as anti-reflective layer was found to exhibit promising results. The stacking of these three materials with appropriate thickness in a systematic manner, viz. Cu/TiAlCrN/TiAlN/AlSiN with high Cr and Al contents in TiAlCrN and TiAlN layers, respectively, yielded high absorptivity ($\alpha_{AM1.5}=0.91$) and low emissivity ($\epsilon=0.07$) values corresponding to an attractive optical selectivity (α/ϵ) of 13. The open air high temperature performance of the optimized multi-layer structure was also investigated in the temperature range 27–700 °C and the stability of the coatings at high temperatures with respect to optical properties was established.

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1. Introduction

In recent years, with exponentially increasing energy demands, there has been a spurt in global efforts to efficiently harness solar energy. Among the various approaches that appear to hold promise, concentrated solar power generation (CSPG) is of major interest [1]. The CSPG process involves three steps: (a) concentration of sun-light on to a Heat Collecting Element (HCE), (b) effective conversion of concentrated solar energy to thermal energy and (c) utilization of thermal energy for steam generation. The conversion efficiency of concentrated solar energy to thermal energy is reported to largely depend on the design of the HCE comprising the receiver tube, as well as the heat exchange fluid [1,2]. The choice of the heat exchange fluid is dictated by the operating temperature range, which can extend up to 380 °C when diathermic oil is used and up to 550 °C by use of molten salts [1,3]. The more frequently used receiver tube materials in HCE have been Copper (Cu) and Stainless Steel (SS) [1,2]. However, these receiver tube materials have inherently poor optical selectivity, θ (θ =absorptivity, $\alpha_{AM1.5}$ /emissivity, ϵ) in the wavelength range of interest (0.315 μm for α and 2.5–25 μm for ϵ) for solar thermal applications and, hence, demand a surface treatment to enhance solar selectivity.

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In recognition of the above, it is of considerable interest to the solar energy community to economically produce high-temperature air-stable solar selective coatings on a variety of receiver tube materials. So far, several selective coatings involving different material combinations and deposition methods have been reported [2,4–7]. The range of coatings investigated has spanned black paints (Cr, Co, Mo, Ni, W, etc.) [2,7–9], particulate coatings, composite films (Cr, Co, Mo, Ni, W, Ag, Au, Pt, etc. in a ceramic matrix like Al₂O₃, SiO₂, etc.) [3,10–12], absorbing semiconductor or metal tandems, and multi-layer structures (Al/WAIN/WAIN/AIN, TiAlN/TiAlON/Si₃N₄, TiAlSiN/TiAlSiON/SiO₂, etc.) [13–15]. In case of multi-layer structures, the material selection, individual layer thickness and their sequence of stacking plays a vital role in deciding the solar selective properties [16,17]. While some of the above coatings have yielded attractive properties, their use has primarily been restricted to 350 °C because of their limited stability upon exposure to high temperature ($T > 350$ °C). However, with the recent advancements in energy concentrator and energy transferring fluid technologies [1], high temperature stable solar selective coatings on receiver tubes have gathered particular momentum in an effort to realize further enhanced efficiencies and long term durability.

Development of solar selective coatings stable in air at temperatures greater than 350 °C demands materials that possess high thermal and structural stability, as well as deposition techniques that afford excellent adhesion with the substrate, with minimal inherent stress. With respect to high temperature stability, various

materials have been tried using different deposition techniques [18–21]. Among them, the refractory nitrides, oxides and nano-composite materials have shown extreme high temperature stability, hardness and inertness [21,22]. Recently, TiAlN thin films with their variable optical properties (obtainable by changing film composition and microstructure) have been reported to be good candidates for photo thermal applications [23,24].

Conventionally, most of the solar selective coatings reported in earlier studies have been deposited using Chemical Vapor Deposition (CVD) or Physical Vapor Deposition (PVD) routes [25,26]. Growing thin films by CVD route demands expensive and toxic precursors, besides involving elevated temperature processing, thereby not making it an ideal choice as a green energy technology. In contrast, the relatively clean and low temperature PVD processes are a better option. Among the many available PVD routes, use of magnetron sputter deposition for solar selective coatings has been most widely reported [2,3,8,13]. Although RF sputtering has been preferred by virtue of its better productivity compared to DC sputtering, it is known to be a very slow and expensive process [13,26].

Notwithstanding the above, little research attention [27] has been devoted to exploring use of the well established and industrially viable cylindrical Cathodic Arc Deposition (*c*-CAD) process, which can potentially yield highly adherent and uniform dense films over large areas, with precise control over composition and thickness, for development of solar selective coatings. In contrast to the planar CAD techniques, the cylindrical cathodes in the *c*-CAD technique can be rotated in a controlled manner at a pre-defined speed, thereby enabling excellent target utilization and efficient cooling of the cathode. The cylindrical geometry and effective cooling of the cathode also result in minimal droplet formation, unlike the conventional planar CAD techniques [28,29].

In the present study, the authors have attempted to formulate a novel functional multi-layer coating, comprising refractory nitrides and nanocomposite materials, on Cu and SS substrates using the *c*-CAD route, which is known to also facilitate development of super hard nanocomposite coatings. Prominent results from this comprehensive study are reported herein.

2. Experimental details

Multi-layer solar selective coatings comprising super hard composite nitride layers of TiAlCrN, TiAlN and AlSiN were developed on Copper (Cu) and Stainless Steel (SS) substrates using a cylindrical Cathodic Arc Physical Vapor Deposition (*c*-CAPVD) system available in the authors' laboratory (Model: π 300, PLATIT). 99.99% pure cylindrical Cr, Ti, Ti_{0.33}Al_{0.67} and Al_{0.82}Si_{0.18} cathodes and high purity nitrogen gas were used for all deposition experiments. In order to ensure contamination free coatings, the deposition chamber was heated to a temperature of 500 °C and then evacuated to a base pressure of 4×10^{-7} mbar prior to the deposition cycle. As a part of pre-coating substrate preparation, all the substrates were first polished using a diamond suspension to achieve a good surface finish (Ra:0.02 μ m) and then cleaned thoroughly using ultrasonic treatment successively in alkaline and acetone medium. Argon ion etching was also carried out within the vacuum chamber prior to deposition.

Preliminary experiments were first conducted to determine suitable functional materials to serve as IR reflector/absorber, absorber and anti-reflector, as well as to identify their sequence in the multi-layer structure eventually proposed to be designed. All known high temperature (> 550 °C) stable nitride coatings possible with the above four available cylindrical cathodes (namely TiN, CrN, TiAlN, TiAlCrN and AlSiN) were grown on Cu substrates to an identical thickness of ~ 180 nm, at a constant

substrate temperature (T_{Sub} : 400 °C) and bias voltage (V_{Sub} : –50 V). The composition of any given film (e.g., Ti content in TiAlN) could be manipulated by appropriately controlling the relevant cathode current. The above thickness was chosen to achieve maximum absorptance in the visible region (~ 600 nm) of the solar spectrum based on considerations of destructive interference. Since TiAlN is a well reported absorber material with a refractive index (n at 632 nm) ~ 2.5 , its optical properties were taken as a first approximation to determine that a 180 nm thickness would generate destructive interference conditions. All the grown films were studied for their phase constitution as well as optical, electrical and microstructural properties. Spectral emissivity (for room temperature studies) and thermal emissivity (> 100 °C) studies were carried out using an FT-IR spectrophotometer (Model: Vertex 70 FTIR, Bruker), equipped with an integrating sphere and special accessories like a black body furnace and a high temperature sample cell unit. From the spectral emittance data emissivity weighted to black body emission is calculated using the relation [6]

$$\varepsilon = \frac{\int_{\lambda_1}^{\lambda_2} \varepsilon_{\lambda} E_{b\lambda} d\lambda}{\int_{\lambda_1}^{\lambda_2} E_{b\lambda} d\lambda}$$

where ε : emissivity (weighted to black body emission spectrum), ε_{λ} : emission intensity of the sample at wavelength λ , $E_{b\lambda}$: black body emission intensity at wavelength λ , and $(\lambda_1-\lambda_2)$ is the wavelength range of interest for emissivity.

In the case of the thermal emittance (at > 100 °C) measurement, emission values of both the black body furnace and the samples were measured at different temperature ranging from 100 °C to 500 °C. From the data the thermal emissivity is obtained by taking the ratio of thermal emission from sample to the black body. The absorptance values were measured using a UV–visible–NIR spectrophotometer (Model: 5000, CARY Varian). For absorptance measurements, the reflectance was first determined and then, using Kirchhoff's law for opaque materials ($\alpha=1-R$), the actual absorptance was calculated. The absorptance values presented herein for all preliminary studies are values averaged over the 0.3 μ m to 1.5 μ m wavelength range. As the UV–visible–NIR spectrophotometer cannot give the absorptivity value directly, for generalization of the absorptance values, the best optimized sample absorptance values were weighted to solar AM 1.5 using the following well-known equation [6]:

$$\alpha_{AM\ 1.5} = \frac{\int_{\lambda_1}^{\lambda_2} \alpha_{\lambda} G_{\lambda} d\lambda}{\int_{\lambda_1}^{\lambda_2} G_{\lambda} d\lambda}$$

where $\alpha_{AM1.5}$: absorptivity weighted to solar spectrum, α_{λ} : absorptance at wavelength λ , G_{λ} : solar radiation intensity at particular wavelength λ and $(\lambda_1-\lambda_2)$ is the wavelength range of interest for absorptance.

The refractive index and extinction coefficient were obtained using a spectroscopic ellipsometer (Model: M-2000V, J. A. Woollam Co. Inc.) in the wavelength range 350–1000 nm. The theoretical absorption coefficient (α') values of individual materials were calculated from the extinction coefficients using the equation $\alpha' = 4\pi k/\lambda$. The electrical resistivity measurements were made using linear four point probe technique at 10 different current values and their average has been reported. The phase constitution of the films was determined using X-ray diffraction (Model: X'pert Pro, PANalytic) with copper K_{α} radiation while X-ray photo electron spectroscopy was used to study the surface oxidation resistance.

Following initial studies aimed at evaluating relevant optical and electrical properties of mono layer nitride films, the film compositions that can best serve as absorber, anti-reflector and IR reflector/absorber layers were identified. The above layers were

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