



Design and characterization of solar absorbing multilayer stack based on Al/Cr-N-O / SiO₂ layers



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ABSTRACT

Design, preparation and characterization of a solar selective absorbing coating based on chromium oxynitride are reported in this contribution. All the layers were deposited by reactive magnetron sputtering in a horizontal in-line sputtering system. The optical constants of individual layers were obtained by spectroscopic ellipsometry measurement and fitting. The absorbing multilayer stack including, from substrate to air interface, Al infrared reflective layer/ (Cr/CrN_x//CrN_xO_y) composite absorbing layer/ SiO₂ antireflective layer were then designed and fabricated based on their optical properties and thickness optimization. This absorbing coating is characterized by the simple and stable reactive magnetron sputter coating process with no need for precise control of single layer thickness and reactive gas partial pressure. A solar absorptance more than 95% and thermal emissivity less than 4% for operation temperature of 100 °C were obtained by analyzing the measured spectra data. After aging test in air at 250 °C for 200 h, the performance criterion (PC) was below 1%, and the deterioration mechanism was further analyzed combined with XPS depth profile.

1. Introduction

Solar selective absorbing coatings (SSACs) have attracted much attention for their application in solar collector [1]. In order to obtain the high solar-thermal conversion efficiency, the solar absorbing coating should absorb much of the incident solar energy (in the wavelength from 0.3 μm to 2.5 μm) while losing very little heat by re-radiation (at the wavelength greater than 2.5 μm). Therefore, the absorbing coating should be designed to have solar absorptance (α) as high as possible and thermal emissivity (ε) at an operating temperature as low as possible, simultaneously.

Up to now, one of mainly commercially introduced solar absorbing multilayer stack consists of at least two TiN_xO_y absorbing layers with different x, y values, and SiO_x, Si₃N₄ or Al₂O₃ AR layer, and is claimed to achieve the values of α ~ 95% and ε ~ 4% with a optimized collector operation temperature of approx. 100 °C [2]. There are some other multilayer stacks with very similar coating compositions, replacing titanium in the above mentioned TiN_xO_y by aluminum [3], chromium [4], nickel [5], and a composition of two or more metal (for example, AlTi [6], WAl [7]); and the optical selectivity of these coatings does not improve obviously; and thermal stability can be enhanced if two or more metal is applied in metal nitride/oxynitride absorbing layers [6,7].

The solar absorbing coatings based on above-mentioned various metal nitrides/oxynitrides are usually coated by magnetron sputtering from the pure metal or alloy targets in a reactive atmosphere containing nitrogen or oxygen. The component of nitride or oxynitride mainly determines the optical properties of SSACs, and it is sensitive to the reactive sputtering process, especially partial pressure of the reactive gas. Therefore, a minor change in partial pressure of the reactive gas could cause the obvious change in solar absorptance, thermal emissivity, and even surface color of coatings [3–8]. And they have to be controlled precisely during sputtering coating in order to obtain the desirable optical selectivity of SSACs. For large area coating, there are disadvantages such as complex coating process and high demands of accuracy and stability for the control system of coating equipment. But, up to now, there is no study about how to optimize the optical properties of SSACs, and then facilitate coating process of SSACs based various metal nitrides or oxynitrides.

In this work, firstly, the stoichiometry of chromium nitride and oxynitride has been tailored by varying the deposition conditions, thus, an optical constant database including various refractive index and extinction coefficient belonging to different CrN_x and CrN_xO_y are obtained. Afterwards, using this optical constant database, a solar selective absorbing coating based on Al infrared reflective layer/ Cr-N-O composite absorbing layer/ SiO₂ antireflective layer is presented, in

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which the Cr-N-O composite absorbing layer consists of a metal absorbing sublayer Cr, a metal nitride absorbing sublayer CrN_x, and a metal oxynitride absorbing sublayer CrN_xO_y. This multilayer stack is characterized by the following features: 1) an excellent optical selectivity; 2) the simple and stable reactive magnetron sputter coating process with no need for precise control of single layer thickness and reactive gas partial pressure.

2. Experimental details

2.1. Sample preparation and characterization

The single layer CrN_x, CrN_xO_y, Al, SiO₂ films were deposited on glass substrates, using a horizontal in-line sputtering system evacuated with a cryogenic pump. The different planar cathodes were installed on the slots on the top lid of the chamber. During deposition, the substrates moved back and forth under the cathodes for multiple passes. The film thickness was controlled by the substrate moving speed and the number of passes. A silicon target doped with aluminum was used in our study in order to improve the ductility to reduce the target cracking during deposition.

The detailed deposition parameters of various CrN_x, CrN_xO_y films with different stoichiometry are shown in Tables 1 and 2, and those of Al and SiO₂ films are given in Table 3.

For all the samples, the optical measurements in the wavelength range of 0.3–2.5 μm, including transmittance and reflectance modes, were performed using a Hitachi U4100 spectrophotometer equipped with an integrating sphere. The reflectance measurements were performed at an incidence angle of 8° using Al mirror as reference, and the reflectance data were corrected accordingly with the Al-reference reflectance curve. The solar absorptance, α, is defined as a weighted fraction between absorbed radiation and incoming solar radiation and obtained using either calculated or experimentally measured spectral reflectance data $R(\lambda)$ and ASTM AM1.5D solar spectral irradiance, $A(\lambda)$, according to [9]:

$$\alpha = \frac{\int_{0.3\mu\text{m}}^{2.5\mu\text{m}} A(\lambda)[1 - R(\lambda)]d\lambda}{\int_{0.3\mu\text{m}}^{2.5\mu\text{m}} A(\lambda)d\lambda} \quad (1)$$

And the infrared reflection spectra (2.0–48 μm) measurement were performed using a Fourier Transform Infrared (FTIR) spectrometer (SENSOR 27, BRUKER) with an incidence angles of 12°. The infrared emissivity (ε) is a weighted fraction between emitted radiation and the Planck blackbody radiation at a specific temperature and calculated from [9]:

$$\varepsilon = \frac{\int_{2\mu\text{m}}^{48\mu\text{m}} E_T(\lambda)[1 - R(\lambda)]d\lambda}{\int_{2\mu\text{m}}^{48\mu\text{m}} E_T(\lambda)d\lambda} \quad (2)$$

where $E_T(\lambda)$ is the spectral blackbody emissive power, at 100 °C in our study, and $R(\lambda)$ is the measured IR spectral reflectance.

Table 1

Deposition parameters of the CrN_x single layers.

Deposition parameter	Value
System base Pressure	< 6.67 × 10 ⁻⁴ Pa
Process Pressure	0.40 Pa
Target material	Chromium
Target size	457 mm(length) × 127 mm (width) × 6 mm (thickness)
Target to substrate distance	70 mm
Power (Pulse DC)	1500 W
Substrate temperature	Room temperature
Substrate	6 mm soda-lime glass
Substrate moving speed	0.4 m/min
Argon flow	50 sccm
Nitrogen flow	0, 5, 10, 15, 20, 25, 30, 35, 40, 45, 50 sccm, respectively

Table 2

Deposition parameters of the CrN_xO_y single layers.

Deposition parameter	Value
Argon flow	50 sccm
Nitrogen flow	50 sccm
Oxygen flow	3, 4, 5, 6, 7, 8, 9, 10, 12 sccm, respectively

The other deposition parameters are the same as those in Table 1.

Table 3

Deposition parameters of the Al and SiO₂ single layers.

Deposition parameter	Al	SiO ₂
Target material	Aluminum	Silicon(30 wt%Al)
Power (Pulse DC)	1200 w	2000 w
Oxygen flow	0	14 sccm
Argon flow	50 sccm	30 sccm

The other deposition parameters are the same as those in Table 1.

For the solar absorbing multilayer stack, the accelerated ageing tests in air were performed in a muffle furnace at temperature of 250 °C and 400 °C for different duration. The performance criterion PC value ($PC = \Delta\alpha - 0.5\Delta\varepsilon$), in which $\Delta\alpha = \alpha(\text{unaged}) - \alpha(\text{after testing})$ and $\Delta\varepsilon = \varepsilon(\text{unaged}) - \varepsilon(\text{after testing})$, was calculated to describe the influence of the changes in the solar absorptance and the emissivity after the accelerated aging test.

The quantitative elemental compositions of the chromium nitride and oxynitride individual layers and the elemental depth profile of the absorbing multilayer stack were obtained by X-ray photoelectron spectra (XPS, PHI Quantera SXM spectrometer). The XPS binding energy was calibrated with C 1s = 284.6 eV, and the Ar⁺ energy of 4 keV with 100 μA ion current was utilized for the layer-by-layer etching.

2.2. SE measurement and fitting

The spectral optical constants of various chromium nitrides and oxynitrides can be determined from spectroscopic ellipsometry (SE) measurement and fitting of their individual layer coated glass [10,11].

SE measurements were performed over 300–2500 nm at 5 nm interval in air at ambient temperature using a Variable Angle Spectroscopic Ellipsometer (V-VASE, J. A. Woollam Co.) at incidence angles of 55°, 60°, 65° and 70°, respectively. In order to avoid rear-side reflection of the glass substrates during SE measurement, single layer samples sputtered on 6 mm soda-lime glass were used. The measured ellipsometric data (Psi, Delta) and transmission spectra were curve fitted to a model of suitable dielectric function for different films using WVASE32 software (J. A. Woollam Co.). The optical constants and the thickness of the films were then obtained at the minimum RMS for the fitting. The thickness of films was also measured by a surface profiler (Dektak 150, Veeco Instruments, Inc.) to verify the thickness values obtained from ellipsometry.

In order to analyze the obtained ellipsometric data of different film coated glass in our study, an optical model including a surface roughness layer (hereafter referred to as SR) and bulk layer is constructed as in Fig. 1, where, d_{SR} and d_{bulk} indicate the thicknesses for surface roughness and bulk layer of different film, respectively. The surface of every coated sample can be looked as no scattering surface, for the reason that its corresponding SR thickness in SE fitting is not more than 4 nm, which is much shorter than the incident optical wavelength (0.3–2.5 μm).

We modeled the dielectric function of the surface roughness layer as a 50/50 vol% mixture of a film bulk layer and voids, applying Bruggeman effective-medium theory.

The film bulk layer is regarded as a homogeneous material which is isotropic and uniform. The dielectric function of different bulk layer can

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