Solar Energy Materials and Solar Cells 176 (2018) 93–99

Contents lists available at ScienceDirect

Solar Energy Materials and Solar Cells

journal homepage: www.elsevier.com/locate/solmat

Optical properties and failure analysis of ZrC-ZrO_x ceramic based spectrally selective solar absorbers deposited at a high substrate temperature

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ARTICLE INFO

Keywords:
Solar absorber
Optical properties
Failure analysis
Substrate temperature
Ultra-high temperature ceramics

ABSTRACT

A tandem layer structured ZrC-ZrO_x/Al_2O_3 coatings are deposited onto stainless steel (SS) substrates by the sputtering method for solar selective absorbing. The ZrC-ZrO_x and Al_2O_3 layers work as an absorptance layer and an antireflectance layer, respectively. The substrate temperature has an important effect on the optical properties of the coating. With a high substrate temperature (300 °C), the SS/ZrC-ZrO_x/Al_2O_3 coatings exhibit a relatively high absorptance of 0.92 and a relatively low emittance of 0.12. The composition, structure, optical properties and surface morphology of the coatings are characterized using SEM, EDS, XPS, Raman, UV–vis–NIR spectrophotometry and Fourier transform infrared spectroscopy. Detailed failure analysis of the SS/ZrC-ZrO_x/Al_2O_3 coatings in vacuum and air is conducted.

1. Introduction

Zirconium carbide (ZrC), one of the well-known Ultra-high temperature ceramics (UHTCs), combines the characteristic properties of metal and ceramic due to a mixture of ionic, covalent and metallic bonding. As one of the important high temperature structure materials, ZrC has unique combination of chemical, structure and mechanical properties, such as high melting point, very high hardness, excellent thermal shock resistance, good wear resistance and high electrical conductivity. Due to an excellent combination of structure compatibility and mechanical properties, it works as a promising candidate for applications as cutting tools, wear resistant components and crucibles in the mechanical industry, especially nuclear reactor core material [1–5].

Besides ultra-refractoriness and favorable mechanical and chemical characteristics, ZrC ceramic also shows intrinsic spectral selectivity, which can be explained by two contributions from in-band (or Drude) and inter-band (or Lorentz) according to the rigid band model of the electronic structure of the atoms [6]. The high melting point of ZrC together with good thermal-mechanical properties and potential spectral selectivity, makes it as a promising materials for solar receivers and solar absorbers. For solar receivers, Pierrat studied the oxidation of ZrC-20 vol% $MoSi_2$ in the temperature range 1800–2400 K in air, in order to partially reproduce the operating conditions of a high temperature receiver for concentrated solar radiation [7]. Sani investigated the intrinsic spectral selectivity of different carbide samples of UHTCs for new applications as sunlight absorbers in tower solar plants [8,9]. Sani also studied the microstructure, mechanical and optical properties of dense zirconium, hafnium and tantalum carbides as a function of the sintering method (high pressure or pressureless), implying use of 10 or 20 vol% of $MoSi_2$ as sintering aid [10]. For solar absorbers, ZrC was reactively sputtered in N_2 to deposite ZrC_xN_y solar absorber coating on silver (Ag), which exhibited an absorptance of 0.88 and an emittance of 0.03. The agglomeration of silver layer at 350 °C degrades infrared reflecting surface properties, thus causing an enhancement in emittance [11]. Bonnot et al. studied the reflectance spectra of sputtered ZrC_x and ZrC_xN_y thin films by means of their electronic structure and by the nature of the bonding. By adjusting the carbon and nitrogen contents, the optical profile of ternary compounds of transition metal can be tailored in a wide range between those of the binary compounds [12]. Lazarov et al. has fabricated the ZrO_x/ZrC_x/Zr absorber-reflector tandem solar absorbers by sputtering with Zr as an infrared reflector. The solar absorbers exhibited high spectral selectivity with α/ε (20 °C) = 0.90/0.05 and good thermal stability on stainless-steel and quartz substrates up-to 600 °C and 800 °C in vacuum, respectively [13,14]. Recently, Fang et al. developed the Zr-Al(Si)-C quaternary system high temperature solar absorbers by adding Al(Si) into ZrC with a spectral selectivity of 2.8 (0.71/0.25). The incorporation of Al and or Si can greatly improve the oxidation resistance and fracture toughness [15]. Usmani et al. fabricated the ZrO_x/ZrC-ZrN/Zr absorber-reflector

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https://doi.org/10.1016/j.solmat.2017.11.018
Received 23 April 2017; Received in revised form 10 October 2017; Accepted 13 November 2017

tandem solar absorbers on stainless steel (SS) and copper (Cu) substrates using DC/RF magnetron sputtering system. Through optimized the nitrogen flow rate, the solar absorbers exhibited an absorptance of 0.88 and 0.85 and an emittance of 0.04 and 0.1 on stainless steel and copper substrates, respectively. Thermal studies showed high temperature stability at 700 °C and 600 °C in vacuum for these solar selective coatings on SS and Cu substrates, whereas at or below 200 °C in the air [16]. Then they studied the impact of corrosion on microstructure and mechanical properties of ZrO_x/ZrC-ZrN/Zr absorber-reflector tandem solar selective structures [17].

Notwithstanding these advances, the rational design and facile fabrication of ZrC based solar absorbers with enhanced spectral selectivity still remain as a challenge. More importantly, the failure analysis such as degradation mechanism of optical properties should be conducted. In this work, we demonstrate a more simple and economical strategy to deposite SS/ZrC-ZrO_x/Al_2O_3 solar absorbers with a high substrate temperature of 300 °C, which exhibit a high absorptance of 0.92 and a low emittance of 0.12 (82 °C). The structure, optical properties and thermal stability are investigated in detail. We also provide a more detail failure analysis particularly on optical properties of SS/ZrC-ZrO_x/Al_2O_3 solar absorber coatings in vacuum and air.

2. Experimental

2.1. Coating preparation

The SS/ZrC-ZrO_x/Al_2O_3 coatings were fabricated using a commercial magnetron sputtering system (Kurt J. Lesker PVD75, USA). High purity ZrC (99.99%) and Al_2O_3 (99.99%) targets (diameter = 76.2 mm) were used for the deposition of the coatings. The SS substrates were metallographically polished to obtain a smooth surface and ultrasonically cleaned with isopropyl alcohol and acetone to remove grease and dust particles on the surface of the substrate. An DC sputtering technique was used to deposite the ZrC layer. Then Al_2O_3 was deposited using RF sputtering technique. The complete deposition process was performed in an argon plasma environment with a nitrogen flow 33 sccm. For comparison, the substrate temperatures is 100 °C, 200 °C and 300 °C, respectively. The optimized process parameters for the deposition of the tandem absorber are listed in Table 1.

2.2. Characterization

The SS/ZrC-ZrO_x/Al_2O_3 coatings were heat-treated under vacuum in a tubular furnace at temperatures in the range of 400–700 °C for 5 h and 100 h, respectively. The surface morphologies were observed by ultra-high resolution scanning electron microscopy (SU8200, Tokyo, Japan). The chemical bonding state and chemical composition of the coating were defined via X-ray photoelectron spectroscopy (XPS, equipped with a standard monochromatic AlKα source of 1486.6 eV, ESCALAB 210, VG scientific Ltd., UK). The XPS binding energy data was calibrated with respect to the C1s signal of ambient hydrocarbons (C-H and C-C) centered at 284.8 eV. Changes in the chemical composition of the solar absorber coatings as a result of heating were measured using Raman spectroscopy (DILOR-JOBIN-YVON-SPEX).

Reflectance spectra in the wavelength interval 0.3–2.5 µm was measured on a Perkin Elmer Lambda 950 UV/Vis/NIR spectrometer with an integration sphere (module 150 mm), while reflectance spectra in the wavelength interval 2.5–25 µm was obtained on a Bruker TENSOR 27 FT-IR Spectrometer, equipped with an integrating sphere (A562-G/Q) using a gold plate as a standard for diffuse reflectance.

Table 1
Optimized process parameters for the deposition of the tandem layer SS/ZrC-ZrO_x/Al_2O_3 spectrally selective solar absorber coating.

Layer	Sputtering method	Ar flow rate (sccm)	Power density (W/cm^2)	Thickness (nm)	Substrate temperature (°C)	Operating pressure (Pa)
ZrC	DC	33	6.58	240	300	1.03
Al_2O_3	RF	33	6.14	84	300	1.03

3. Energy performance and chromaticity

3.1. Energy performance

The normal α_s and ε_T values were calculated using Eqs. (1) and (2). The solar absorptance α_s is theoretically defined as a weighted fraction between absorbed radiation and incoming solar radiation.

$$\alpha_s(\theta, \lambda) = \frac{\int_{0.3}^{2.5} [1 - R(\theta, \lambda)] I_s(\lambda) d\lambda}{\int_{0.3}^{2.5} I_s(\lambda) d\lambda} \quad (1)$$

where λ is wavelength, R (λ) reflectance and Is (λ) direct normal solar irradiance. It is defined according to ISO standard 9845-1, normal radiance, AM1.5. Normal thermal emittance ε_T is equally a weighted fraction but between emitted radiation and the Planck black body distribution, I_b (λ, T), at temperature T.

$$\varepsilon_T(\lambda, T) = \frac{\int_{2.5}^{25} [1 - R(\theta, \lambda)] I_b(\lambda, T) d\lambda}{\int_{2.5}^{25} I_b(\lambda, T) d\lambda} \quad (2)$$

Accordingly, thermal emittance values of the samples were denoted as ε_{82} in this work when these were obtained at 82 °C.

3.2. Chromaticity

Based on the description of the XYZ color system by the international commission on illumination (CIE) in 1931, the tristimulus values X, Y, Z are computed from the measured or simulated spectral power distribution data P(λ) [18].

$$X = \int_{380}^{780} P(\lambda)\bar{x}(\lambda) d\lambda \quad (3)$$

$$Y = \int_{380}^{780} P(\lambda)\bar{y}(\lambda) d\lambda \quad (4)$$

$$Z = \int_{380}^{780} P(\lambda)\bar{z}(\lambda) d\lambda \quad (5)$$

where λ is the wavelength. In this work, $P(\lambda) = D_{65}(\lambda)R(\lambda)$, where D_{65} is a standard illuminant and represents the typical daylight. Then the color coordinates x, y, and z are calculated by

$$x = \frac{X}{X + Y + Z} \quad (6)$$

$$y = \frac{Y}{X + Y + Z} \quad (7)$$

$$z = \frac{Z}{X + Y + Z} \quad (8)$$

4. Results and discussion

4.1. X-ray photoelectron spectroscopy

With an aim to investigate the chemical composition and chemical state, ZrC and ZrC/Al_2O_3 layers are deposited on Si substrates and characterized by XPS. Fig. 1(a) and (b) shows the high resolution XPS core level spectra of the ZrC layer after sputtering with a 3 keV Ar^+ ion beam for 300 s. As shown in Fig. 1(a), the Zr3d core level spectrum exhibits two peaks centered at 180.9 eV and 183.3 eV, which corresponds to ZrC and ZrO_x bonds, respectively [19,20]. Thus, the component of the absorption layer is ZrC-ZrO_x. The carbon core level