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# Annealing effect on the optical properties and interdiffusion of MgO/Zr/MgO multilayered selective solar absorber coatings

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#### Abstract

Selective solar absorber coatings based on MgO/Zr/MgO multilayered coatings were prepared onto Zr coated stainless steel substrates using e-beam evaporation. It was found that MgO/Zr/MgO multilayered solar absorber coatings exhibited a good spectral selectivity of 0.91/0.12. The effect of annealing on the optical properties and interdiffusion between the layers was investigated. Annealing the multilayered coating in air up to 300 °C did not show significant change on the spectral selectivity as well as on the interdiffusion between the layers indicating the coating's thermal stability up to that temperature. Annealing in air at 400 °C leads to decrease in solar absorptance and an increase in thermal emittance considerably, which was due to the interlayers appeared between MgO and Zr layers, oxidation of Zr layers, and change in both thickness and composition of the layers as investigated by RBS analysis. The MgO/Zr/MgO MSSACs were found to be thermally stable up to 250 °C in air for 24 h. © 2015 Elsevier Ltd. All rights reserved.

Keywords: Spectral selectivity; Optical properties; Thermal stability; Interdiffusion

#### 1. Introduction

Due to the global economic growth and human development, energy demand continues to increase. Hence, looking for a clean and reliable energy is crucial. Solar energy, one of the renewable resources, is infinite and available anywhere on earth. Hence to harness this infinite energy, various technologies such as solar heating, solar photovoltaic and concentrated solar power (CSP) systems are involved. The latter one converts the incoming solar

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http://dx.doi.org/10.1016/j.solener.2015.07.022 0038-092X/© 2015 Elsevier Ltd. All rights reserved. radiation into heat energy before generating electricity through thermodynamic process (Zheng et al., 2015). Efficient conversion of solar radiation into heat in CSP systems requires a selective solar absorber surface. The ideal behavior of a selective solar absorber surface is having high solar absorptance in the solar spectrum region  $(0.3-2.5 \,\mu\text{m})$  and low thermal emittance in the infrared region (above 2.5 µm) Nuru et al., 2012a, 2012b; Feng et al., 2015. The solar absorptance describes that fraction of the solar flux which is absorbed by the surface, and the thermal emittance provides an index to the capacity of a surface for radiating thermal power (Thornton, 1982). This implies a step-like reflectance spectrum with reflectance (R) = 0 in the solar spectrum region and

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R = 1 in the infrared region. The ratio of solar absorptance to thermal emittance is called spectral selectivity ( $\alpha/\varepsilon$ ) Nuru et al., 2014; Selvakumar and Barshilia, 2012, which is used to evaluate the spectral selectivity of a solar absorber material. In addition to these optical performance requirements, a selective solar absorber coating has to withstand high temperatures when exposed to vacuum or air (Nuru et al., 2012, 2015). To achieve these characteristics, various concepts and materials have been explored.

Several multilayered selective solar absorber coating (MSSAC) using alternative layer of metals (Pt, Ti, Cr, W, Mo, Ta, Al and Ni) and dielectrics  $(Al_xO_y, SiO_2, HfO_2,$  $Al_2O_3$ , and  $Cr_2O_3$ ) have been investigated as selective solar absorber coatings for mid and high temperature CSP applications (Nuru et al., 2014; Barshilia et al., 2008; Selvakumar et al., 2010; Tsai et al., 2014; Thornton and Lamb, 1982; Nuru et al., 2014; Barshilia et al., 2009). Nuru et al. reported that  $Al_xO_y/Pt/Al_xO_y$  MSSAC was thermally stabile in air up to 450 °C for 24 h with good spectral selectivity of 0.96/0.08 (Nuru et al., 2014). Barshilia et al. revealed that  $Cr_xO_y/Cr/Cr_2O_3$  MSSAC was thermally stable in air up to 250 °C for 250 h with spectral selectivity of 0.898/0.11 (Barshilia et al., 2008). HfO<sub>x</sub>/Mo/HfO<sub>2</sub> MSSAC was thermally stable in air up to 400 °C for 1 h with spectral selectivity of 0.91/0.07 (Selvakumar et al., 2010). Tsai et al. revealed that  $Al_xO_v/Ni/Al_xO_v$  MSSAC was thermally stable in air up to 400 °C for 12 h with spectral selectivity of 0.93/0.04 (Tsai et al., 2014). Thornton et al. investigated the thermal stability of Al<sub>2</sub>O<sub>3</sub>/M/Al<sub>2</sub>O<sub>3</sub>/R-type MSSAC with M layers of Cr, Ni, Mo, and Ta layer (Thornton and Lamb, 1982). They found that multilayered coatings with reactively sputtered Al<sub>2</sub>O<sub>3</sub> layers change absorptance value in the range of 300–450 °C in air or vacuum, but radio frequency (RF)sputtered Al<sub>2</sub>O<sub>3</sub> layers were thermally stable in the range of 500-600 °C and 650-700 °C in air and vacuum, respectively.  $Al_xO_v/Pt/Al_xO_v$  MSSAC with a tantalum diffusion barrier was also found to be thermally stable up to 700 °C in air (Nuru et al., 2014). Al<sub>x</sub>O<sub>v</sub>/Al/Al<sub>x</sub>O<sub>v</sub> MSSAC deposited on to Cu substrates exhibited good thermally stability up to 400 °C in air and the coatings, which were deposited onto Mo substrates were thermally stabile up to 800 °C in vacuum (Barshilia et al., 2009). Most of the MSSACs degrade at higher temperature because of interdiffusion between the layers and oxidation.

Maziere-Bezes et al. reported magnesium oxide (MgO) based selective solar absorber cermet with good optical properties (Maziere-Bezes and Valignat, 1983). Due to its high thermal stability, high melting point, and outstanding electrical insulating properties with good thermal conductivity, it is believed that MgO is ideal to use as selective solar absorber coatings for high temperature CSP systems. Based on these peculiar properties, we have reported recently a new MgO/Zr/MgO MSSACs, which is thermally stable in vacuum up to 400 °C for 2 h with a good spectral selectivity of 0.92/0.09 (Nuru et al., 2015). However, the study of heat treatment in air for the MgO/Zr/MgO

MSSAC has not been studied. In this paper, we report the effect of annealing in air on the optical properties and interdiffusion between the layers of MgO/Zr/MgO MSSACs. Thermal stability study under air is crucial in case the vacuum is breached (Kennedy, 2002).

### 2. Experimental details

MgO/Zr/MgO MSSACs were deposited onto stainless steel (SS) substrates using a 3 kW high vacuum e-beam evaporation system at room temperature. The experimental details have been described in detail elsewhere (Nuru et al., 2015). The thickness and composition of the as-deposited and heat treated samples was studied by Rutherford backscattering spectroscopy (RBS). RBS was performed using a 1.57 MeV He+ beam with the sample mounted on a three-axis goniometer. The composition and thickness of the films were obtained by fitting the RBS spectra using the RUMP simulation software (Doolittle, 1985). The error bar of the thickness was  $\pm 3$  nm.

Spectral reflectance was measured with a Cary 5000 UV–VIS–NIR spectrophotometer of Varian, Inc. model internal DRA-2500, in the wavelength range of 0.3–2.5  $\mu$ m. The solar absorptance was calculated from the measured reflectance data and weighted by solar irradiance using standard the AM1.5 solar spectrum in the above wavelength range. Thermal emittance spectra were acquired by an Emissometer model AE1, which has a precision of  $\pm$ 0.01 emittance units.

In order to study the thermal stability of the MSSAC, the coatings were heat treated in air in a furnace (Elite Thermal Systems Limited model TSH12/50/610-2416CG) at temperatures in the range of 100–500 °C, and 150–350 °C at an increment of 100 °C for 2 and 24 h, respectively at each temperature. The temperature was ramped from room temperature to the desired temperature at a rate of 7 °C/min, and subsequently cooled down at a rate of 10 °C/min. The accuracy of the set temperature was  $\pm$ 5 °C.

## 3. Results and discussion

It is well known that the thermal stability of selective solar absorber coatings is crucial as the absorber can degrade with time at the operating temperatures when exposed to vacuum or air, which shrinks the life time and eventually leads to failure. For this reason, MgO/Zr/MgO MSSACs which was deposited onto Zr coated SS was heat treated in air at different temperatures and durations. Fig. 1 shows the reflectance spectra of MgO/Zr/MgO MSSACs. The corresponding solar absorptance and thermal emittance values are shown in Fig. 2 and Table 1. At 100 and 200 °C, minimal change in the solar absorptance and thermal emittance was observed. For the MSSAC which was annealed at 300 °C, only a slight change in the solar absorptance and thermal emittance was seen. This indicates that the MgO/Zr/MgO MSSAC

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