ORIGINAL PAPER

High‑Entropy Oxide Solar Selective Absorber

Yi‑Cheng Lin1 · Fitri Nur Indah Sari1 · Siang‑Yun Li1 · Jyh‑Ming Ting1

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Abstract

Sputter-deposited high-entropy materials, including high-entropy alloy (HEA) and high-entropy oxide (HEO), are demonstrated for use as selective solar absorber coatings (SSCs). Multi-layer SSC consists of CrFeCoNiAl HEA as the IR refector layer, (CrFeCoNi)O medium-entropy oxide as the frst layer absorber, and (CrMnFeCoNi)O HEO as the second layer absorber. The efects of phase and elemental concentration of the second layer absorber on the optical properties and thermal stability are addressed. The (CrMnFeCoNi)O HEO hematite has better spectral selectivity than the (CrMnFeCoNi)O HEO spinel. Meanwhile, from the X-Ray difraction and transmission electron microscopy analyses of the annealed samples, the (CrMnFeCoNi)O HEO spinel exhibits better thermal stability than (CrMnFeCoNi)O HEO hematite. This work provides guidance to create an efective solar absorber.

Graphical Abstract

(CrMnFeCoNi)O hematite solar absorber exhibits better spectral selectivity than (CrMnFeCoNi)O spinel. (CrMnFeCoNi) O spinel exhibits better thermal stability than (CrMnFeCoNi)O hematite. The thermal instability of the spinel originates from the metal difusion.

Keywords High-entropy oxide · Solar absorber coating · Spinel · Hematite · Thermal stability

 \boxtimes Jyh-Ming Ting jting@mail.ncku.edu.tw

¹ Department of Materials Science and Engineering, National Cheng Kung University, Tainan 7010, Taiwan

Introduction

There are various green, renewable energy systems that convert the solar energy into electricity directly, such as solar photovoltaic cell [[1](#page-6-0)], or indirectly, such as solar thermal power systems [[2,](#page-6-1) [3\]](#page-6-2). One of the most promising solar thermal power systems is the concentrating solar power (CSP) system, involving the use of a solar selective absorber to convert the sunlight into thermal electric power. To maximize the efficiency, high absorptance (α) in the solar spectrum range and low thermal emittance (ε) in the infrared range (IR) are required. Depending on operation temperature, solar selective absorber can be grouped into low temperature (T <100 °C), mid temperature (100 °C < T < 450 °C), and high-temperature $(T > 450 \degree C)$ absorbers [\[4](#page-6-3)]. In the context of high-temperature solar selective absorber, Pyromark 2500, Ciemat, and spinel oxide-pigmented coatings are known as the state-of-the-art coatings. Pyromark 2500 is silicon-based paint that has high thermal stability up to 700 °C. However, the value of α starts to degrade at operating temperatures >700 °C in air from 90 to 88% due to phase transformation and spallation [\[5,](#page-6-4) [6\]](#page-7-0). Multi-layer Ciemat coatings having Pt IR layer and two difusion layers is also thermally stable up to 500 °C with α of 93% [\[7](#page-7-1)]. However, this coating degrades as the temperature increases to above 650 °C in air due to the degradation of Pt IR layer. Moreover, spinel oxide-pigmented coating exhibits a high thermal stability up to 750 °C with α of 93% [\[8](#page-7-2)]

Currently, high-temperature solar selective absorbers are being sought since a higher operation temperature leads to more thermal power to electricity conversion. Metal-dielectric composite or the so-called cermet is a conventional high-temperature solar selective absorber. A cermet consists of metal nanoparticles embedded in a dielectric matrix. For examples, $Cr-Cr₂O₃$ cermet is known as the black chrome [[9\]](#page-7-3). To enhance solar absorption, a sandwiched structure consisting of an IR refector in the bottom, a solar selective absorber in the middle, and an anti-refective (AR) layer at the top is often used. In such a solar selective coating (SSC), the IR refector refects the thermal emission from the metallic substrate and the AR layer helps to enhance the light absorption. Moreover, the solar absorber itself is often multi-layered for improved solar absorption. Hightemperature multi-layered SSCs include SS substrate/Cr IR refector/low oxygen content Cr-AlOx/mid-oxygen content $Cr-AlO_x$ layer/high oxygen content $Cr-AlO_x$ layer, giving α =0.91 and ε =0.225 after annealing at 700 °C for 2 h in air [\[10\]](#page-7-4), SS/TiN/alternating TiAlON and TiAlN layers, giving α = 0.788 and ε = 0.173 after annealing at 600 °C for 50 h in air [[11\]](#page-7-5), SS/Mo/HfO_x/Mo/HfO₂, exhibiting absorption and emittance of 0.874 and 0.15, respectively, after annealing at 525 °C in air $[12]$ $[12]$.

In the past few years, a new class of materials, namely, high-entropy materials, has emerged at the center stage of materials research, thanks to the discovery of high-entropy alloy in 2004 [[13\]](#page-7-7) and the report of high-entropy oxide (HEO) in 2015 [[14](#page-7-8)]. HEMs have been shown to exhibit a number of excellent characteristics. High-temperature structural stability between 850 and 1000 °C has been demonstrated in, for example, (MgCoNiCuZn)O [\[14\]](#page-7-8) and between 450 and 850 °C in, for example, $(CoCrFeMnNi)₃O₄$ [[15](#page-7-9)]. $(Yb_{0.2}Y_0,Lu_{0.2}Sc_{0.2}Gd_{0.2})_2Si_2O_7$ has good corrosion resistance in water–vapor environment and displays excellent phase stability from room temperature to 1300 $^{\circ}$ C [[16](#page-7-10)]. $(Mg_{0.2}Co_{0.2}Ni_{0.2}Cu_{0.2}Zn_{0.2})O$, (Li, Mg, Co, Ni, Cu, Zn)O, and (Zn, Fe, Ni, Mg, Cd)Fe₂O₄ have been shown to exhibit huge dielectric constants [[17](#page-7-11), [18\]](#page-7-12). Along with the demonstration of these characteristics, HEOs have been prepared using methods, such as solid-state reaction [[14](#page-7-8), [19](#page-7-13)], hydrothermal synthesis [\[20,](#page-7-14) [21](#page-7-15)], solvothermal synthesis [[22\]](#page-7-16), solution combustion synthesis [[15](#page-7-9)], nebulized spray pyrolysis [[23\]](#page-7-17), and reverse co-precipitation [[24\]](#page-7-18). The resulting materials are powders or nanoparticles. It appears that there are and only limited studies reported thin-flm HEOs obtained using a PVD process [\[25](#page-7-19)]. In the meantime, in the past few years, there are several works on high-entropy materials for solar absorber material, including AlCrTaTiZrN [[26](#page-7-20)], MoNbHfZrTi [[27\]](#page-7-21), HfNbTaTiZrN [\[28](#page-7-22)], MoNbHfZrTi [\[29](#page-7-23)], AlMo0.5NbTa0.5TiZrNx [[30\]](#page-7-24), and HEO, has been demonstrated for solar absorber. It is seen that almost no study demonstrate the use of HEO for solar absorber coating.

In this work, we have explored the use of sputter-deposited HEAs and HEOs for use SSCs. The IR refector layer is a CrFeCoNiAl HEA, and the multi-layered absorber consists of a medium-entropy oxide (MEO) of (CrFeCoNi)O and HEO of (CrMnFeCoNi)O. Relation among the composition, structure, and optical property is investigated. The resulting SSCs have also been evaluated for the thermal stability. In the meantime, we have also investigated the efect of two diferent HEO crystal structures, e.g., HEO spinel and hematite, for the degradation mechanisms. This work provides a guidance for optimizing oxide material for high-temperature solar absorber coating.

Experimental

CrFeCoNiAl HEA, (CrFeCoNi)O MEO, and (CrMn-FeCoNi)O HEO were deposited on (100) Si and 316L SS substrates using a RF co-sputtered deposition method. The target-to-substrate distance was fixed at 130 mm and the deposition chamber was evacuated to lower than 5× 10−6 Torr and then back flled with 20-sccm Ar before the deposition. The working pressure was 5×10^{-3} torr and the substrate was rotated at 5 rpm. High-purity (99.99%)

Ar and $O₂$ gasses were used as working gas and reactive gas, respectively. For the CrFeCoNiAl IR refector (designated as IRr), an Al target (99.995%) and a composite target of Cr, Fe, Co, Ni (99.995%) were used. The RF powers for the Al and composite targets were 80 and 120 W, respectively. For the frst absorber layer of (CrFeCoNi)O MEO (designated as A1), the composite target was used. The power was 100 W and $O_2/Ar = 2$ sccm/20 sccm. Manganese oxide (MnO) target (99.995%) and the composite target were used to deposit the second absorber layer of (CrMnFeCoNi)O HEO. Two such HEOs were made. One was obtained at a MnO target power of 100 W, (CrFeCoNi) alloy target power of 80W, and $O_2/Ar = 2$ sccm/20 sccm, giving sample A2a. The other was obtained at a MnO target power of 80 W, (CrFeCoNi) alloy target power of 100W, and $O_2/Ar = 7$ sccm/20 sccm, giving sample A2b. The substrate was externally heated at 800 °C for the deposition of all oxides. In contrast, no external heating was applied to the substrate for the deposition of the HEA. A matrix summarizing the deposition conditions is given in Table S1 in the Supporting Information. For the thermal stability test, as-deposited samples were post-annealed in air at diferent temperatures of 600, 700, and 800 °C for 2 h (heating rate = 6.67 °C/min).

The thickness of coating was determined using proflometer and feld emission scanning electron microscopy (SEM, JOEL6701). The chemical composition was examined using Energy-dispersive X-ray spectroscopy (EDS, JOEL6701). The refractive index and extinction coeffcient were investigated using ellipsometer (J.A. Woollam /M2000-DI). The crystal structure was studied using glazing angle X-ray difraction (GIXRD, Bruker D8-Discover). The refectance from 300 to 2500 nm wavelength was measured using UV/Vis/NIR spectrophotometry equipped with an integrated sphere (LAMBDATM 950, PerkinElmer). The solar absorptance (α) was calculated as follows using Eq. [\(1\)](#page-2-0) [[31\]](#page-7-25), where E_s is the intensity of incident solar light, A is the absorbance, and R is the refectance.

$$
\alpha = \frac{\int_{300}^{2500} E_s(\lambda) A(\lambda) d\lambda}{\int_{300}^{2500} E_s(\lambda) d\lambda} = \frac{\int_{300}^{2500} E_s(\lambda) [1 - R(\lambda)] d\lambda}{\int_{300}^{2500} E_s(\lambda) d\lambda}.
$$
 (1)

Emission was measured using Emissionmetry (Emissionmeter K3, Optosol GmbH). It consists of an integrating sphere, glow bars as the sources of thermal radiation, and 3 detectors, which are sensitive for 3.9-, 5.1-, and 10-μm wavelengths. Emissionmetry gave thermal emittance (ε) at room temperature that at 100 $^{\circ}$ C was calculated. Thermal emittance was calculated as follows using Eq. ([2](#page-2-1)) [[31](#page-7-25)], where $E_{100\degree C}$ is the black body radiation at 100 °C.

$$
\varepsilon = \frac{\int_{2500}^{10000} A(\lambda) E_{100 \circ C}(\lambda) d\lambda}{\int_{2500}^{10000} E_{100 \circ C}(\lambda) d\lambda}.
$$
 (2)

The structure properties of the annealed coatings were investigated using GIXRD and transmission electron microscopy (HR-TEM, JEOL JEM-2100F).

Results and Discussion

SEM analysis shows that the surface morphology of the HEA IRr is granular (Fig. [1a](#page-3-0)). Similar surface morphology was found for the MEO and HEO absorber layers, as shown in Fig. [1](#page-3-0)b for HEO. From the cross-section, it is seen that all layers have columnar structure, which is typical structure obtained using sputter deposition. This morphology with surface texture is beneficial for light absorption, thus increasing solar absorption and thermal ability [\[32\]](#page-7-26). All the coating thicknesses are very uniform, as shown in Fig. [1c](#page-3-0) and d for the IRr and A1 layers, respectively. The HEA IRr has a BCC structure (JCPDF #47-1126) [[33\]](#page-7-27), as shown in Fig. [2](#page-3-1). EDS analysis shows that the composition of the HEA is Cr/Fe/Co/Ni/Al=10.7/25.1/6.0/5.1/23.2 in at %. The A1 MEO layer is a spinel oxide (JCPDF #73-1856), having Cr/ Fe/Co/Ni=9.7/9.8/12.8/67.7 in at %. The A2a HEO also has a spinel structure (JCPDF #84-0482). Meanwhile, the A2b HEO has a hematite structure (JCPDF #84-0311). The chemical compositions of A2a and A2b are Cr/Mn/Fe/Co/ Ni = 4.6/47.1/5.2/7.9/35.3 and 8.5/25.5/7.1/11/47.9, respectively. The diferent chemical compositions in the A2a and A2b are due to the diferent power used for the MnO and CrFeCoNi composite. Sputtering power is related to the kinetic energy of the target atoms. A higher sputtering power results in higher surface difusions once these atoms are adsorbed on the substrate surface [[34\]](#page-7-28). As a result, A2a has larger amount of Mn than that of A2b layer. This is due to A2a was deposited at higher sputtering power of 100 W, while A2b was deposited at smaller sputtering power of 80 W. Likewise, the Cr, Fe, Co, and Ni concentrations are higher in A2b layer due to its higher sputtering powers of CrFeCoNi target.

The reflective indexes of these single layers are first presented. The refective indexes at 500 nm of the single layers are 3.50, 2.62, 2.35, and 2.32 for IRr, A1, A2a, and A2b, respectively (Fig. [3](#page-3-2) and Table S1). The reflective index gradually decreases from the IRr to A1 and then to A2. Meanwhile, the extinction coefficient at 500 nm of the single layers are 2.78, 0.6, 0.62, and 0.86 for IRr, A1, A2a, and A2b, respectively. As a result, SSCs consisting of IRr/A1/ A2a and IRr/A1/A2b various layers are examined. Figure [4a](#page-4-0) shows the refectance spectra of various single and multilayer SSCs. The addition of layers gives low refectance in

Fig. 2 GIXRD patterns of as-deposited coatings on Si substrate

the solar spectrum, indicating higher α. Comparing the IRr/ A1/A2a and IRr/A1/A2b, the IRr/A1/A2a has slightly better α of 0.81 vs 0.78, as shown in Fig. [4b](#page-4-0). However, the ε of the IRr/A1/A2b is slightly smaller than IRr/A1/A2a, 0.111 vs 0.130. Spectral selectivity (α/ε) of both multilayer SSCs is thus determined. The IRr/A1/A2b has α /ε of 7, which is higher than that of IRr/A1/A2a (6.2). To understand the better performance of the IRr/A1/A2b, the refectance spectra of single layers A2a and A2b were obtained, as shown in Figure S1. The single layer A2b exhibits higher α /ε of 6.6, compared to that of A2a (6). Moreover, the absorption of

Wavelength (nm) 600 800 1000 1200 1400 1600 Refractive index 0.0 0.5 1.0 1.5 2.0 2.5 3.0 Extinction coefficient 0.0 0.5 1.0 1.5 2.0 2.5 3.0 A1 (n) A2a (n) A2b (n) A1 (k) A2a (k) A2b (k)

Fig. 3 Reflective index and extinction coefficient of as-deposited coatings on Si substrate

the thin flm occurs due to two mechanisms, one is intrinsic absorption, which is characterized by the extinction coefficient of the film and the other one is interference-induced absorption. The A2b has a higher extinction coefficient of 0.86 than A2a (0.6), indicating its higher absorption capability.

The thermal stability of the SSCs was thus investigated at diferent temperatures of 600, 700, and 800 °C for 2 h in air. Figure [5](#page-4-1) shows that the solar absorptance and emittance of IRr/A1/A2a coating remain the same at 600 °C. The decrement of spectral selectivity is observed with the rising temperature. On the other hand, IRr/A1/A2b coating

Fig. 4 a Refectance spectra and **b** solar absorptance and emittance of as-deposited single and multiple layer coatings on SS substrate

Fig. 5 a Refection spectra and **b** absorptance and emittance of as-deposited IRr/A1/A2a SSC on SS substrate and post-annealing at 600, 700, and 800 °C in air

exhibits a higher solar absorptance and smaller emittance at 600 °C (Fig. [6\)](#page-5-0). The spectral selectivity decreases at a higher temperature of 700 °C and signifcantly decreases at 800 °C.

The SSCs show optical degradation after annealing at≥700 °C. Various post-analysis was performed to understand the phase, morphology, and elemental changing after annealing treatment. XRD analysis was frst performed on the post-annealing A2a and A2b coating layers to understand the phase stability after annealing. The crystal structure of the A2a after post-annealing at 600 and 700 °C remains the same as before annealing, which is spinel oxide (Fig. [7a](#page-5-1)). This indicates the structural stability of A2a. It was reported that the HEO spinel has high thermal stability due to its small chemical difusion coeffcient at low-pressure oxygen (high temperature) [[35,](#page-7-29) [36](#page-7-30)].

Meanwhile, A2b shows crystalline structure changes with the annealing temperature. The structure transforms from hematite for as-synthesized coating to spinel oxide for coating after post-annealing at 600 and 700 °C, as shown in Fig. [7](#page-5-1)b. Furthermore, the crystallinity of the spinel oxide becomes better with increasing temperature. Thus, the phase transformation in A2b accounts for thermal degradation. Moreover, the result also confrms that spinel oxide has better phase stability than hematite oxide. Due to there is no phase transformation in the A2a, the post-TEM analysis was performed on IRr/A1/A2a-700 to further investigate the root cause of the degradation. It was found that the thickness of the A1 layer of IRr/A1/A2a-700 was wider as compared to the as-deposited IRr/A1/A2a, 25.1 vs 8.4 nm (Fig. [8](#page-6-5)). On the other hand, the thickness of the

Fig. 6 a Refectance spectra and **b** absorptance and emittance of as-deposited IRr/A1/A2b SSC on SS substrate and post-annealing at 600, 700, and 800 °C in air

Fig. 7 XRD patterns of as-deposited SSC and post-annealing at 600 and 700 °C in air for 2 h on SS substrate

A2a layer after annealing at 700 °C becomes shrinkage. EDS analysis was performed to understand the elemental concentration change due to difusion at high temperature. It is seen that A2a and IR layers show insignifcant change after annealing at 700 °C. Meanwhile, the A1 layer shows signifcant change in elemental composition after annealing at 700 °C. Almost all of the metal concentration decreases after annealing at 700 °C, except Al, as shown in Table [1](#page-6-6). The reduction of the Cr, Mn, Fe, Co, and Ni concentration in the A1 layer of IRr/A1/A2a might be due to the difusion into either IRr or A2a layers. It is noted that the Al concentration in A1 signifcantly increases after annealing at 700 °C, from 9.6 to 31.7%. This indicates the difusion of the Al from the IRr layer toward A1. This can be explained since the melting point of Al is 660 °C, thus annealing at 700 °C triggers its difusion. The difusion of Al also can be seen from EDS mapping (Figures S2 and S3). Furthermore, after annealing, it is obvious that the Cr in the IRr layer is not uniformly distributed, forming aggregation. From the post-XRD, -TEM, and -EDS analyses, the degradation of the spectral selectivity of the IRr/ A1/A2a is due to the chemical structural instability in the A1 layer and aggregation of Cr in the IRr layer.

Fig. 8 TEM images of **a** as-deposited IRr/A1/A2a and **b** IRr/A1/A2a after post-annealing at 700 °C in air for 2 h on SS substrate

Conclusion

A1 layers

In this work, sputter-deposited high-entropy materials were demonstrated for use as SSCs. Multi-layer SSC consists of CrFeCoNiAl HEA as the IR refector layer, (CrFeCoNi) O MEO as the frst layer absorber, and (CrMnFeCoNi)O HEO as the second layer absorber. The multi-layered structure enhances the solar absorptance and reduces the emission. Moreover, the phase and elemental concentration of the second absorber layer affect the spectral selectivity and thermal stability. The (CrMnFeCoNi)O HEO hematite has better spectral selectivity than (CrMnFeCoNi)O HEO spinel due to its higher extinction coefficient. Meanwhile, the (CrMnFeCoNi)O HEO spinel exhibits better thermal stability than (CrMnFeCoNi)O HEO hematite due to its phase and structural stability.

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Data Availability Data are available on request.

Declarations

Conflict of interest On behalf of all authors, the corresponding author states that there is no confict of interest.

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