Enhanced thermal stability of ZrAlSiN cermet-based solar selective absorbing coatings via adding silicon element

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A B S T R A C T

Thermal stability is an important performance for the application of the photothermal conversion coating at high temperature. In this article, we find an effective method to improve the thermal stability of the cermet coating based on the MeAlN (Ti, Zr, Hf and so on) as absorbing layer. Adding a little Si element (~ 5 at.%) into the Zr0.3Al0.7-N (mainly absorbing layer) can modify the microstructure and form the nanocrystalline ZrAlN with amorphous-phase around it. This microstructure can suppress the spinodal decomposition of c-ZrAlN, and it benefits to decrease the nitrogen diffusion. Then, the thermal stability of coating can be improved. The novel Cu/Zr0.32Al0.63Si0.05N/Zr0.21Al0.73Si0.06N/Al31Si3O60N6 cermet coating exhibits the excellent thermal stability than the Cu/Zr0.37Al0.63Si0.05N/Zr0.27Al0.73Si0.06N/Al31Si3O60N6 cermet coating at 600 °C in vacuum. It indicates that adding a little Si (~ 5 at.%) into the cermet coating based on the MeAlN (Ti, Zr and Hf and so on) as absorbing layer coating is an effective way to improve the thermal stability.

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Introduction

Photothermal conversion technology is an important way to harvest the solar energy directly. In photothermal conversion system, solar collector is a device which absorbs solar radiation, converts it to thermal energy, and delivers the thermal energy to a heat transfer medium [1]. As we all know, solar selective absorbing coatings can enhance the photothermal conversion efficiency of solar collector obviously. For the parabolic trough collector (PTC) applied in concentrating solar power system, solar selective absorbing coating services at high temperature (above 400 °C) under vacuum. Certainly, it is important for solar selective absorbing coating to process the good chemical inertness and thermal stability at elevated temperature. In PTC system, mineral or synthetic oil is generally utilized as heat-transfer fluid (HTF). Its limitation is the low operating temperature (around 400 °C) and hydrogen gas generated by the thermal decomposition [2]. Molten salt (such as 60% KN03-40% NaNO3) is a more stabley HTF. Additionally, it can be operated at higher temperature (more than 550 °C) [3–5]. The higher operating temperature is expected because the higher temperature results in the higher Cannot efficiency of power plants [6]. For PTC used molten salt as the heat-transfer fluid, a new solar selective absorbing coating which has high thermal stability and good optical properties needs to be developed.

Many coatings based on the different designed principle and physical mechanism have been developed [7]. Among those coatings, cermet coatings based on the bi-sublayer cermet film structure are demonstrated to process high absorbance and low emittance [8,9]. It becomes the preferred design system. Currently, the nitride of transition metals, such as TiN, CrN, ZrN, HfN, NbN, TiON, CrON, etc., is often chose as absorbing unite because of high thermal stability [10–16]. Generally, the nitride of transition metals (metal-like materials, absorbing unit) combined with AlN (dielectric material) is used as absorbing layer in the cermet coatings. The TiAIN is the commonly used material, whereas the c-MeAIN (Me = Ti, Zr, and Hf and so on) is a metastable phase. At high temperature, the c-MeAIN phase will transform into the c-MeN and c-AlN by the spinodal decomposition [17–21]. Then, the metastable c-AlN will transform into the stable phase h-AlN finally. During this process, the Al–N bond will break, which results in the escape and
loss of nitrogen atom [22]. The introduction of silicon into the MeAlN system is an effective way to modify the microstructure and properties. The formation of the amorphous Si3N4 on the grain boundary will refine grain and form the nanocrystalline MeAlN/amorphous-phase nanocomposite structure which amorphous Si3N4 boundary layers encapsulate nanocrystalline TiAlN grains [23,24]. This structure can suppress or retard the process of spindal decomposition and the formation of stable h-AlN at the high temperature [23,25–27]. This can suppress the escape of N, which can improve the thermal stability of cermet coatings based on the nitride of transition metals as absorbing units.

In this article, ZrN is chosen as the absorbing units. A little Si element (~5 at.%) is introduced into the absorbing layer to improve the thermal stability of coating. A new cermet coating of Cu/Zr0.32Al0.63Si0.05N/Zr0.21Al0.73Si0.06N/Al31Si3O60N6 was optimization designed. Its optical properties, microstructure, and thermal stability are studied.

Experimental

The Cu, Zr0.32Al0.63Si0.05N, Zr0.21Al0.73Si0.06N, Al31Si3O60N6 films, and Cu/Zr0.32Al0.63Si0.05N/Zr0.21Al0.73Si0.06N/Al31Si3O60N6 coatings were deposited on Si(111) and glass (soda lime glass) substrates by ISB700 system. The substrates were ultrasonically cleaned with acetone and ethanol at room temperature. The single layer (Cu, Zr0.32Al0.63Si0.05N, Zr0.21Al0.73Si0.06N, Al31Si3O60N6) deposited on glass (soda lime glass) was used for the optical analysis. The films and coatings were deposited on Si(111) was for studying microstructure and thermal stability. A copper target (99.99% purity), an Al90Si10 compound target (90 at.% aluminum and 10 at.% silicon), and a zirconium target (99.9% purity) were sputtered by argon ion and oxygen ion) generated by a Kaufman ion source bombarded the substrate to synthesize the Zr3Si5N12-x-y-z and Al31Si3O60N6 sub-layers. To avoid the charge accumulation on the surface of the insulation substrate (such as Si(111) or glass), neutral filament at exit of reaction ion source was used to neutralize the nitrogen or oxygen ion. The base pressure was 2.0 × 10⁻⁴ Pa. The detail parameters were listed in Table 1. The solar selective absorbing coating has to work under a good vacuum (~10⁻² Pa) at high temperature according to the concentrated ratio of solar radiation [28]. Considering the temperature fluctuation during service, the annealing temperature is 400°C and 600°C, respectively. The coating deposited on Si(111) substrate was used to carry out the annealing experiment in vacuum (5.0 × 10⁻⁵ Pa) ambient at 400°C or 600°C for different hours.

The optical constants of Zr0.32Al0.63Si0.05N, Zr0.21Al0.73Si0.06N, and Al31Si3O60N6 were measured by spectroscopic ellipsometry (Type: M-2000UJ). The detail information about the fitting process was given in Supplementary. Based on the optical constants of the single layer, the reflectance spectra of the cermet coatings with four-layer structure was optimized and calculated by a commercial optical simulation program (Version Name: TFCalc 3.5.6). The spectral reflectance in the wavelength region from 250 to 2500 nm was measured at the incidence angle of 8° by using the UV-Vis-NIR spectrophotometer equipped with an integrating sphere (Type: SHIMADZU UV3600). The reflectance in the range of 2.5–25 μm was measured by Fourier transform infrared spectrophotometer (Type: Vertex80). The total solar absorptance (εsolar) is obtained according to the following formula:

$$α_{solar} = \frac{\int_{250}^{2500} I_{solar}(\lambda)(1 - R(\lambda))d\lambda}{\int_{250}^{2500} I_{solar}(\lambda)d\lambda}$$  \hspace{1cm} (1)

where, $R(\lambda)$ is the measured reflectance from 250 nm to 2500 nm, and $I_{solar}(\lambda)$ is the ASTM AM1.5D solar spectral irradiance.

Thermal emittance (εthermal(T)) at the specific temperature is determined by the formula as follow:

$$ε_{thermal}(T) = \frac{\int_{0}^{\infty} I_{blackbody}(\lambda)(1 - R(\lambda))d\lambda}{\int_{0}^{\infty} I_{blackbody}(\lambda)d\lambda}$$  \hspace{1cm} (2)

where, $ε_{thermal}(T)$ is an emittance at the temperature $T$. In our article, temperature is 400°C. $I_{blackbody}(\lambda)$ is the spectral radiation of black body at 400°C according to the Planck’s radiation law.

The microstructure of coatings is investigated by transmission electron microscope (TEM, type: Tecnai G2 F20). The TEM is equipped with a high-angle angular-dark-field detector and X-ray energy-dispersive spectrometer systems. Element distribution in depth was measured by Auger electron spectroscopy (AES, type: ULVAC- PHI 700).

Results and discussion

The refractive index (Fig. 1a) and extinction coefficient (Fig. 1b) of the single film, including Zr0.32Al0.63Si0.05N, Zr0.21Al0.73Si0.06N and Al31Si3O60N6, are shown in Fig. 1. The low mean-squared error (MSE) indicates the obtained optical constants is credible (Fig. S1, Fig. S2 and Fig. S3). In the case of Zr0.32Al0.63Si0.05N film, it is observed that there is an increase of the refractive index with the wavelength, whereas the extinction coefficient initially decreases with the wavelength, and it reaches to the minimum around 400 nm. Then, the extinction coefficient increases with wavelength and reveals a maximum at 1100 nm. These results show the metal-like behavior of Zr0.32Al0.63Si0.05N film [29]. It is the main absorbing layer. For Zr0.21Al0.73Si0.06N film, the refractive index initially reaches to the maximum at the wavelength of ~320 nm; thereafter, it decreases. Its extinction coefficient decreases monotonically. Additionally, the extinction coefficient value is between 0.1 and 1.1.

Table 1

<table>
<thead>
<tr>
<th>Materials</th>
<th>Target</th>
<th>Ar (sccm)</th>
<th>N2 (sccm)</th>
<th>O2 (sccm)</th>
<th>AIE (keV)</th>
<th>ASIE (mA)</th>
<th>AES (eV)</th>
<th>AIC (mA)</th>
<th>Time (min)</th>
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</thead>
<tbody>
<tr>
<td>Al0.31Si0.69O0.6N</td>
<td>Al0.31Si0.69O0.6N</td>
<td>5</td>
<td>2</td>
<td>4</td>
<td>2.7</td>
<td>100</td>
<td>200</td>
<td>50</td>
<td>14</td>
</tr>
<tr>
<td>Zr0.32Al0.63Si0.05N</td>
<td>Zr0.32Al0.63Si0.05N</td>
<td>5</td>
<td>6</td>
<td>2.7</td>
<td>100</td>
<td>200</td>
<td>50</td>
<td>3</td>
<td></td>
</tr>
<tr>
<td>Zr0.21Al0.73Si0.06N</td>
<td>Zr0.21Al0.73Si0.06N</td>
<td>5</td>
<td>6</td>
<td>2.7</td>
<td>100</td>
<td>200</td>
<td>50</td>
<td>13</td>
<td></td>
</tr>
<tr>
<td>Cu</td>
<td>Cu</td>
<td>5</td>
<td>0</td>
<td>2.7</td>
<td>100</td>
<td>200</td>
<td>30</td>
<td>10</td>
<td></td>
</tr>
</tbody>
</table>

AIE, AIC, ASIE, and ASIC correspond to argon ion energy, argon ion current, assisted ion energy, and assisted ion current. Al0.31Si0.69 target which contains 90 at.% aluminum and 10 at.% silicon.
Based on above results, the Zr0.21Al0.73Si0.06N exhibits an intermediate behavior (i.e. between metallic and dielectric) [30]. Both refractive index and extinction coefficient of Al13Si3O60N6 film decrease with wavelength. The extinction value is almost zero, which displays the dielectric behavior in nature. The low refractive index contributes to the low front surface reflectance (Fig. 2). The Zr0.21Al0.73Si0.06N and Al31Si3O60N6 is 100 nm, 65 nm, 15 nm, and 75 nm, respectively. Then, the Cu/Zr0.32Al0.63Si0.05N/Zr0.21Al0.73Si0.06N/Al31Si3O60N6 cermet coating with approximately optimized thickness is prepared on Si(111) and glass. Comparing the theoretical and experimental reflectance spectra of coating, a good agreement between theoretical can be observed (Fig. 2). The measured d-spacing from the diffraction role of γ-Al2O3 (200) and c-ZrN(220) (PDF database file 02-0956). The measured d-spacing deviating with the theoretical values is because of the substitution of zirconium atoms by aluminum atoms which has the smaller atom radius than the atom radius of zirconium [32]. The high resolution transmission electron microscope (HRTEM) analysis of the single sublayer is carried out to investigate the sublayer microstructure. The crystalline phase of c-ZrAlN (Zone A in Fig. 3b) and amorphous phase around it can be featured in the Zr0.32Al0.63Si0.05N sublayer. In the case of Zr0.21Al0.73Si0.06N and Al31Si3O60N6 sub-layer. The amorphous structure can be noted from Fig. 3d. Based on the above the results, the diffraction roles are from the crystalline phase in the Zr0.32Al0.63Si0.05N sublayer.

As a comparison, the microstructure analysis of the Cu/Zr0.3Al0.7N/Zr0.3Al0.8N/Al34O60N6 cermet coatings demonstrates that the Zr0.3Al0.7N sub-layer is composed of crystalline phase of h-AlN and c-ZrAlN with a little of the amorphous phase, whereas, the Zr0.2Al0.8N and Al34O60N6 sub-layer are amorphous (Fig. S4) [30]. Adding 5% silicon into the Zr0.3Al0.7N sublayer modifies the microstructure and forms the nc-ZrAlN/amorphous-phase nanocomposite [23,24]. The silicon element has little influence on the microstructure of Zr0.21Al0.73Si0.06N and Al31Si3O60N6 sub-layer.

The formation Gibbs energy at 298 K (ΔGf) of Si3N4, ZrN and AlN is −665.4 kJ/mol [33], −344.0 kJ/mol [34], and −287.0 kJ/mol [35], respectively. The nitrogen atom tends to react with the silicon

Fig. 1. Optical constants of Zr0.32Al0.65Si0.05N, Zr0.21Al0.7Si0.06N and Al13Si3O60N6 films. (a) refractive index. (b) extinction coefficient.

Fig. 2. Schematic diagram of the Cu/Zr0.32Al0.65Si0.05N/Zr0.21Al0.7Si0.06N/Al31Si3O60N6 absorbing coating and corresponding to optimized theoretical and experimental reflectance spectra.
atom to synthesize the Si3N4 at substrate preferentially. The crystallinity of Si3N4 is poor, and Si3N4 tends to form amorphous. In the equilibrium thermodynamic conditions, stoichiometric fcc-ZrN and hcp(b)-Si3N4 are immiscible. The crystalline ZrN is synthesized. Al atom will enter into the ZrN crystal lattice by the interstitial solution, and the ZrAlN solid solution is formed. The amorphous Si3N4 between the nanocrystalline ZrAlN suppresses the grain growth, which will refine the grain size. The nc-ZrAlN/amorphous nanocomposites ("nc" stand for nanocrystalline) is formed. The maximum solid solubility of Al in the ZrN crystal lattice is 0.472 [36]. Additionally, the amorphous Si3N4 at the grain boundary of ZrAlN crystalline cuts off the template effect[37]. The abundant AlN tends to form amorphous structure. For the Zr0.21Al0.73Si0.06N sublayer, the crystal nucleus cannot grow into grain because of the low content of zirconium atom. The crystallinity of Al31Si3O60N6 is poor. Hence, the sublayers of Zr0.21Al0.73Si0.06N and Al31Si3O60N6 are amorphous.

Thermal stability of Cu/Zr0.32Al0.63Si0.05N/Zr0.21Al0.73Si0.06N/Al31Si3O60N6 coatings was performed in a vacuum ambient (5.0 × 10⁻² Pa) using a tubular furnace. Table 3 gives the change of absorptance and emittance of coating after annealing treatment. The absorptance, emittance and photothermal conversion efficiency (\(a/\varepsilon\)) is almost stable. No significant change of the whole reflectance between as-deposited and coating after annealing for 192 h can be noted in Fig. 4a. Whereas, in the case of coating after annealing at 600°C, a continuous increase of emittance is observed. After annealing treatment for 192 h at 600°C, the photothermal conversion efficiency (\(a/\varepsilon\)) decreases from 11.3 to 6.56. Compared with the reflectance of as-deposited coating (see Fig. 4b), the reflectance decreases above ~1900 nm; whereas, it keeps stable below ~1900 nm.

Table 3
Effect of annealing temperature and time on absorptance and emittance of Cu/Zr0.32Al0.63Si0.05N/Zr0.21Al0.73Si0.06N/Al31Si3O60N6 coating.

<table>
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<tr>
<th>Condition</th>
<th>As-deposited</th>
<th>400°C 96 h</th>
<th>400°C 144 h</th>
<th>400°C 168 h</th>
<th>400°C 192 h</th>
</tr>
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<tr>
<td>Absorptance ((a))</td>
<td>0.945</td>
<td>0.946</td>
<td>0.945</td>
<td>0.943</td>
<td>0.943</td>
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<tr>
<td>Emittance ((\varepsilon_{400°C}))</td>
<td>0.084</td>
<td>0.085</td>
<td>0.084</td>
<td>0.085</td>
<td>0.084</td>
</tr>
<tr>
<td>(q/\varepsilon)</td>
<td>11.3</td>
<td>11.1</td>
<td>11.3</td>
<td>11.1</td>
<td>11.2</td>
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</table>

<table>
<thead>
<tr>
<th>Condition</th>
<th>As-deposited</th>
<th>600°C 96 h</th>
<th>600°C 144 h</th>
<th>600°C 168 h</th>
<th>600°C 192 h</th>
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<tbody>
<tr>
<td>Absorptance ((a))</td>
<td>0.945</td>
<td>0.948</td>
<td>0.952</td>
<td>0.953</td>
<td>0.951</td>
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<tr>
<td>Emittance ((\varepsilon_{600°C}))</td>
<td>0.084</td>
<td>0.093</td>
<td>0.107</td>
<td>0.123</td>
<td>0.144</td>
</tr>
<tr>
<td>(q/\varepsilon)</td>
<td>11.3</td>
<td>10.2</td>
<td>8.90</td>
<td>7.75</td>
<td>6.56</td>
</tr>
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</table>
The microstructure and composition analysis was carried out to study the degradation mechanism. The TEM cross-section and HRTEM images of coating after annealing at 600°C are shown in Fig. 5. After annealing treatment at 600°C, the thickness of Zr$_{0.32}$Al$_{0.63}$Si$_{0.05}$N, Zr$_{0.21}$Al$_{0.73}$Si$_{0.06}$N and Al$_{3}$Si$_{3}$O$_{60}$N$_{6}$ keeps stable, and the interface is also distinguishable. The thickness increase of Cu infrared layer is because of the element diffusion between Si(111) substrate and Cu sublayer. The SAED patterns of as-deposited and annealed coating have the same feature. Table 4 gives a detail information of phase analysis. The absorbing and antireflection layer (including Zr$_{0.32}$Al$_{0.63}$Si$_{0.05}$N, Zr$_{0.21}$Al$_{0.73}$Si$_{0.06}$N and Al$_{3}$Si$_{3}$O$_{60}$N$_{6}$ layer) and the structure of the crystalline phase keeps stable. No new crystal phase is observed. To confirm the microstructure further, the HRTEM analysis was implemented. The Zr$_{0.32}$Al$_{0.63}$Si$_{0.05}$N sublayer is composed of nanocrystalline ZrAlN (Zone A in Fig. 5c) with amorphous around it. The Zr$_{0.21}$Al$_{0.73}$Si$_{0.06}$N and Al$_{3}$Si$_{3}$O$_{60}$N$_{6}$ sublayers are still amorphous. The structure of absorbing and antireflection layer is stable.

The element distribution of as-deposited and annealed coating in depth was analyzed by AES. For as-deposited coating (As shown in Fig. 6a), the sublayer is distinguished clearly. The atom concentration rises and drops sharply around interface. After annealing

![Fig. 4. Reflectance spectra of as-deposited and annealed Cu/Zr$_{0.32}$Al$_{0.63}$Si$_{0.05}$N/Zr$_{0.21}$Al$_{0.73}$Si$_{0.06}$N/Al$_{3}$Si$_{3}$O$_{60}$N$_{6}$ coatings: (a) as-deposited (black solid line) and after (red dashed line) annealing at 400°C for 192 h and (b) as-deposited (black solid line) and after (red dashed line) annealing at 600°C for 192 h.](image1)

![Fig. 5. The TEM analysis of annealed coating: (a) TEM cross-section of Zr$_{0.32}$Al$_{0.63}$Si$_{0.05}$N/Zr$_{0.21}$Al$_{0.73}$Si$_{0.06}$N/Al$_{3}$Si$_{3}$O$_{60}$N$_{6}$ coatings, (b) SAED image of Zr$_{0.32}$Al$_{0.63}$Si$_{0.05}$N/Zr$_{0.21}$Al$_{0.73}$Si$_{0.06}$N/Al$_{3}$Si$_{3}$O$_{60}$N$_{6}$ layer corresponding to the area in the image of a, (c) HRTEM image of Zr$_{0.32}$Al$_{0.63}$Si$_{0.05}$N/Zr$_{0.21}$Al$_{0.73}$Si$_{0.06}$N/Al$_{3}$Si$_{3}$O$_{60}$N$_{6}$ interface. TEM, transmission electron microscope.](image2)

<table>
<thead>
<tr>
<th>Table 4</th>
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<tbody>
<tr>
<td>The detail information of diffraction role for as-deposited Zr$<em>{0.32}$Al$</em>{0.63}$Si$<em>{0.05}$N/Zr$</em>{0.21}$Al$<em>{0.73}$Si$</em>{0.06}$N/Al$<em>{3}$Si$</em>{3}$O$<em>{60}$N$</em>{6}$ coating.</td>
</tr>
<tr>
<td>Determination of diffraction rings</td>
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<tr>
<td>s nm$^{-1}$</td>
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<tr>
<td>$r_1$</td>
</tr>
<tr>
<td>$r_2$</td>
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</tbody>
</table>
treatment at 600°C for 192 h, the interface of absorbing layer (including Zr0.32Al0.63Si0.05N and Zr0.21Al0.73Si0.06N) and antireflection layer is still distinct. The element diffuse is little. Whereas, the element diffuse between Cu infrared layer and silicon substrate is serious. For comparison, the AES result of Cu/Zr0.3Al0.7N/Zr0.2Al0.8N/Al34O60N6 coatings after annealing treatment at 600°C for 192 is given in the supporting information (Fig. S5). The nitrogen atoms in the Zr0.3Al0.7N sub-layer diffuse toward into adjacent sub-layer (Cu infrared layer and Zr0.2Al0.8N sub-layer) [30]. Comparing this results of microstructure and element distribution in depth, it can be confirmed that adding a little silicon (5 at.%) modifies the microstructure and enhanced the thermal stability.

In the Zr–Al–N system, the metastable phase of ZrAlN will transform into the stable phases c-ZrN and metastable phase c-AlN by spinodal decomposition at high temperature [17–21]. Then, the metastable phase of c-AlN will transform into the stable phase of h-AlN [18] (Fig. 7a). During this process, the escape and loss of nitrogen atom occurred because the Al–N bond will be broken [22]. The grain boundary as a channel accelerates the diffusion of nitrogen atoms [38,39]. Addition of Si into the ZrAlN can form the nc-ZrAlN/amorphous nanocomposite. This structure can suppress the process of the spinodal decomposition because of the grain refinement and amorphous between the grains [23]. Additionally, the amorphous between the grains blocks the diffuse channel of grain boundary (Fig. 7b). Adding a little Si element into the Zr–Al–N system is an effective way to suppress the element (N or O) diffusion and improve the thermal stability. The low active diffusion energy (0.43 eV) and interstitial diffusion leads to the serious diffusion between silicon and copper at high temperature [40,41]. The formation of the Cu3Si, Cu5Si and Cu15Si4 compound owing to diffusion and reaction between silicon and copper results in the decrease of free electrons in the copper infrared layer [42,43], which decreases the infrared reflection. It leads to the emittance increase of coatings.

**Conclusion**

In this article, a novel Cu/Zr0.32Al0.63Si0.05N/Zr0.21Al0.73Si0.06N/Al31Si3O60N6 cermet coating which has high thermal stability has been designed and deposited. The deposited coating exhibites the high absorptance of 0.948 and the low emittance of 0.084 at 400°C. Adding a little silicon (5 at.%) into Zr0.3Al0.7N cermet layer can change the microstructure and form the nanocrystalline ZrAlN with amorphous phase around it. This structure can suppress the spinodal decomposition of c-ZrAlN. The novel Cu/Zr0.32Al0.63Si0.05N/Zr0.21Al0.73Si0.06N/Al31Si3O60N6 exhibits the excellent thermal...
stability than the Cu/Zr0.3Al2O3N/Zr/NAl2O3NAl2O3N6 cermet coating at same condition. It provides an effective way to improve the thermal stability of the coating based on the MeAlN (Ti, Zr and Hf and so on) as absorbing layer.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.mtphys.2019.100131.

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