

Article

Study on the Corrosion Behavior of YSZ Thermal Barrier Coatings by CMAS Composition

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Abstract

Yttria-stabilized zirconia (YSZ) thermal barrier coatings (TBCs) were fabricated by atmospheric plasma spraying (APS). Three CMAS powders with different compositions (CMAS-1, CMAS-2, CMAS-3) were selected, and corrosion tests were carried out at 1200 °C, 1250 °C, and 1300 °C. The relationships among CMAS viscosity, melting point, and reaction tendency with YSZ coatings were investigated. The results show that CMAS-3 possesses the highest viscosity yet the lowest melting point, CMAS-1 has the lowest viscosity but the highest melting point, and CMAS-2 falls between the two. Quantitative penetration depth measurements reveal that higher viscosity leads to slower infiltration, while a lower melting point enables earlier infiltration onset. At elevated temperatures, all CMAS compositions achieve complete penetration, indicating that the differences in melting point and viscosity become less critical when the temperature is sufficiently high. Corrosion tests reveal that CMAS-3 exhibits the strongest reaction tendency with YSZ coatings, while CMAS-1 shows the weakest. This indicates that the infiltration behavior is governed by a dual control of melting point and viscosity—melting point determines the onset of infiltration, while viscosity controls the penetration rate. This study provides an experimental basis for the design of CMAS-resistant coatings and the evaluation of their environmental adaptability. The key finding is that the melting point plays a dominant role in initiating CMAS infiltration, while viscosity primarily regulates the penetration rate.

Keywords: thermal barrier coatings; CMAS; viscosity; reaction tendency



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1. Introduction

Thermal barrier coatings (TBCs) serve as a critical technology for high-temperature protection of hot-section components in advanced aero-engines and gas turbines. Among relevant materials, yttria-stabilized zirconia (YSZ) stands out as the most widely adopted ceramic coating material owing to its low thermal conductivity, well-matched thermal expansion coefficient and excellent high-temperature phase stability [1,2]. Nevertheless, when engines ingest environmental particulate matter such as sand dust and volcanic ash, corrosion failure induced by calcium-magnesium-aluminum-silicate (CMAS) has emerged as one of the most severe challenges confronting YSZ coatings [3]. Once the service temperature exceeds the melting point of CMAS, melted CMAS rapidly infiltrates through the pores and microcracks of the coating. This process triggers Y³⁺ depletion, t→m

phase transformation and volume expansion, ultimately resulting in spalling failure of the coating [4,5].

To tackle CMAS corrosion, researchers have not only modified YSZ but also actively explored new types of CMAS-resistant coating materials. Rare-earth silicates (such as $\text{RE}_2\text{Si}_2\text{O}_7$ and RE_2SiO_5), as candidate materials for environmental barrier coatings, have attracted extensive research attention regarding their reaction behaviors with CMAS. Researchers from Pennsylvania State University and NASA investigated the CMAS corrosion behaviors of $\text{Yb}_2\text{Si}_2\text{O}_7$ and $\text{Y}_2\text{Si}_2\text{O}_7$ with three distinct Ca/Si ratios (0.635, 0.478, 0.096). The results revealed that the proportion of apatite phase rises with increasing CaO content, and $\text{Yb}_2\text{Si}_2\text{O}_7$ facilitates the crystallization of apatite phase to a greater extent [6]. To further clarify the correlation between the types of RE^{3+} rare-earth cations and CMAS composition, additional studies were carried out to examine how four Ca/Si ratios (0.635, 0.478, 0.277, 0.096) affect the corrosion interactions between CMAS and $\text{RE}_2\text{Si}_2\text{O}_7$ ($\text{RE} = \text{Gd}, \text{Dy}, \text{Er}, \text{Nd}$). The findings demonstrated that $\text{RE}_2\text{Si}_2\text{O}_7$ dissolves into the melted CMAS, precipitating apatite phase ($\text{Ca}_{2+y}\text{RE}_{8+x}(\text{SiO}_4)_6\text{O}_{2+3x/2+y}$) and $\text{RE}_2\text{Si}_2\text{O}_7$ [2]. Scholars from the University of California explored the CMAS corrosion performance of bulk $\text{Y}_2\text{Si}_2\text{O}_7$ against CMAS of varied compositions at 1300 °C and 1400 °C separately. It was found that elevated temperatures accelerate the reaction progress, while corrosion resistance is predominantly governed by CMAS composition, alongside an apatite formation threshold (Ca/Si ratio < 0.25) [7]. Researchers at the Shanghai Institute of Ceramics studied the CMAS corrosion characteristics of $\text{Yb}_2\text{Si}_2\text{O}_7$ under four Ca/Si ratios (1.1, 0.75, 0.64, 0.42). The results indicated that CMAS with the highest Ca/Si ratio generates garnet ($\text{Ca}_3\text{Yb}_2(\text{SiO}_4)_3$) and two apatite phases ($\text{Ca}_2\text{Yb}_8(\text{SiO}_4)_6\text{O}_2$ and $\text{Ca}_4\text{Yb}_6(\text{SiO}_4)_{60}$), whereas CMAS with the lowest Ca/Si ratio forms the $\text{Yb}_2\text{Si}_2\text{O}_7$ phase. Moreover, higher Ca/Si ratios reduce CMAS viscosity and intensify the corrosive attack of CMAS on Yb_2SiO_5 coatings [4].

It should be acknowledged that in addition to YSZ, a variety of advanced coating materials—such as rare-earth zirconates, rare-earth tantalates, rare-earth silicates, and high-entropy ceramics—have been extensively investigated as potential TBC/EBC candidates [6,8,9]. However, YSZ remains the most widely used ceramic topcoat in commercial gas turbine engines due to its well-balanced properties. Although the above-mentioned studies have made important progress in the resistance of novel coating materials to CMAS corrosion, conventional YSZ coatings still face severe CMAS corrosion issues. For a long time, researchers have generally regarded the viscosity of CMAS as the key factor governing its corrosion behavior; the lower the viscosity, the higher the fluidity of the melt, the deeper the penetration, and the more severe the corrosion [10,11]. Nevertheless, viscosity itself is intricately affected by the chemical composition and temperature of CMAS, and the melting point determines the melting state and effective penetration duration of CMAS under a given service temperature [12–14]. Unfortunately, there remains a lack of systematic experimental research on the inherent correlation between the viscosity, melting point and corrosion tendency of CMAS.

For this purpose, typical YSZ coatings were fabricated in this study via atmospheric plasma spraying (APS) technology. Three types of CMAS powders with distinct chemical compositions (denoted as CMAS-1, CMAS-2 and CMAS-3, respectively) were selected; their viscosity and melting points were systematically measured and calculated, and isothermal corrosion tests were carried out at 1200 °C, 1250 °C and 1300 °C. The influence laws of CMAS viscosity and melting point on the corrosion behavior of YSZ were analyzed emphatically.

2. Materials and Methods

2.1. Coating Preparation

The nickel-based superalloys were made into Φ 12 mm $\times$ 3 mm samples. The samples were then washed, dried and sandblasted. The YSZ coating was prepared via APS (Oerlikon Metco, F4-MB, Wohlen, Switzerland). The composition of the YSZ coating is 8 wt.%Y₂O₃-ZrO₂, and the thickness is 1000 μ m. The preparation parameters are shown in Table 1. The prepared samples were treated at 1150 °C/5 min using a tube furnace (Hefei Kejing Materials Technology Co., Ltd., Hefei, China), and then water-quenched to obtain independent YSZ coatings.

Table 1. APS parameters.

Electric Current (A)	Voltage (V)	Spraying Distance (mm)	Argon Flow Rates (L/min)	Hydrogen Flow Rates (L/min)	Powder Feed Air Flow Rates (L/min)	Powder Feed Rates (g/min)
560	59	120	35	6.5	3.5	40

2.2. Corrosion Tests

Based on previous research on engine deposits, CMAS with a Ca/Si ratio of 0.75 was designed [15,16]. However, in actual service, there are also differences in the sediment content of turbine engines, for example, the Ca/Si ratio in high-pressure turbine sediments is as high as about 1.5 [17]. Therefore, three different Ca/Si ratios of CMAS components (molar ratio) were designed, with 47CaO-9MgO-13AlO_{1.5}-31SiO₂ (CMAS-1, Ca/Si ratio of 1.5), 33CaO-9MgO-13AlO_{1.5}-45SiO₂ (CMAS-2, Ca/Si ratio of 0.73), 21CaO-9MgO-13AlO_{1.5}-57SiO₂ (CMAS-3, Ca/Si ratio of 0.36). The CMAS powders were prepared by using analytical reagents CaO, MgO, Al₂O₃, and SiO₂ (Hebei Jincan Metal New Materials Co., Ltd., Hefei, China). The above three types of CMAS powders were mixed in proportion using a ball mill, and the mixed powders were treated in a high-temperature box resistance furnace (Hefei Kejing Materials Technology Co., Ltd., Hefei, China) at 1100 °C/4 h. The CMAS powder was mixed with ethanol to form a suspension, which was then sprayed uniformly onto the coating surface using a spray gun. After drying, the specimens were weighed to determine the CMAS loading, which was controlled at approximately 30 mg/cm². After drying, the samples were placed in a high-temperature box resistance furnace for heat treatment. The heat treatment temperatures are 1200 °C, 1250 °C, and 1300 °C, and the time was 1 h and 10 h.

All corrosion tests were conducted at temperatures where the corresponding CMAS compositions were either fully or partially molten, ensuring that the melt was capable of infiltrating the coating. In addition, all specimens were taken from a single batch of APS-deposited YSZ coating, ensuring identical initial microstructure (including porosity, crack density, and phase composition) for all CMAS corrosion tests. Therefore, the observed differences in infiltration behavior between CMAS-1, CMAS-2, and CMAS-3 are primarily attributed to the variations in CMAS composition (melting point, viscosity, and reactivity) rather than to coating variability.

2.3. Experimental Characterizations

The melting points of three types of CMAS powders were measured via simultaneous thermal analysis (TGA-DSC, STA 449 F5, Netzsch, Selb, Germany) under flowing nitrogen protection. The samples were heated from ambient temperature to 1300 °C at a constant ramp rate of 10 K/min. After corrosion, cross-sectional microstructures of YSZ coatings were observed by scanning electron microscopy (SEM, Phenom XL G2, Phenom, Eindhoven,

The Netherlands) coupled with energy-dispersive X-ray spectroscopy (EDS) for elemental analysis. Phase evolution along coating cross-sections was quantitatively identified using laser Raman spectroscopy (LRS, LabRAM HR Evolution, Horiba, Palaiseau, France) with a 532 nm excitation laser. Raman signals were collected in the wavenumber range of 100–3500 cm^{-1} ; the exposure time was 1 s, and laser power was fixed at 100%. Prior to peak fitting, a Shirley-type baseline correction was applied to each spectrum to remove the fluorescence background. The spectra were then fitted using a mixed Gaussian–Lorentzian (Voigt) function.

3. Results and Analysis

3.1. Corrosion Powders

The XRD patterns and DSC curves of the three types of CMAS powders are shown in Figure 1. It can be seen from Figure 1a, the CMAS-1 powder contains a large amount of CaO and is mainly composed of high-melting-point silicate ((Mg,Ca)SiO₃); the CMAS-2 powder is mainly composed of diopside (Ca(Mg,Al)(Si,Al)₂O₆) and (Mg,Ca)SiO₃; the CMAS-3 powder is mainly composed of Ca(Mg,Al)(Si,Al)₂O₆, calcium feldspar (CaAl₂Si₂O₈), corundum (Al₂O₃) and cristobalite (SiO₂). At high temperatures, Ca(Mg,Al)(Si,Al)₂O₆ reacts with Al₂O₃ to form CaAl₂Si₂O₈ and spinel (MgAl₂O₄). Meanwhile, an eutectic system of CaAl₂Si₂O₈ + MgAl₂O₄ + SiO₂ forms in the presence of abundant excess SiO₂. The melting points of CMAS-1, CMAS-2, and CMAS-3 powders are 1257 °C, 1232 °C, and 1187 °C, respectively (Figure 1b), which is consistent with the compositions of the three types of CMAS powders (Figure 1a).

The viscosity of CMAS melt was calculated using the Giordano model (Equation (1)) [11]. Meanwhile, SiO₂ serves as a network-forming agent, while CaO (and other alkali or alkaline earth oxides such as MgO) is considered a network modifier, resulting in a decrease in CMAS viscosity with increasing CaO/SiO₂ ratio and MgO content. AlO_{1.5}, as an amphoteric oxide, can serve as both a network-forming agent and a network modifier [10,14].

$$\log \eta = A + \frac{B}{T(K) - C} \quad (1)$$

where η is viscosity, A is a constant, and B and C are related to the type and content of oxides.

$$B = \sum_{i=1}^7 [b_i M_i] + \sum_{j=1}^3 [b_{1j} (M_{1j} \cdot M_{2j})] \quad (2)$$

$$C = \sum_{i=1}^6 [b_i N_i] + [C_{11} (N_{11} \cdot N_{21})] \quad (3)$$

where M_s and N_s are the combination of the molar percentages of oxides of B and C , respectively. The specific data can be found in reference [11].

The B and C values of the CMAS-1, CMAS-2, and CMAS-3 powders were calculated to be 865.03 and 843.45, 3339.25 and 739.15, and 5460.01 and 649.75, respectively. The viscosity results of the three types of CMAS powders (Figure 2) were obtained by substituting the results into Equation (1). The viscosities of the three CMAS melts were estimated using the Giordano model for qualitative comparison only. It should be noted that the calculated values serve to illustrate the relative viscosity trend (CMAS-1 < CMAS-2 < CMAS-3), rather than to provide absolute predictions. Moreover, the overall infiltration behavior is governed by the combined effects of melting point, liquid fraction, and viscosity, rather than by viscosity alone.

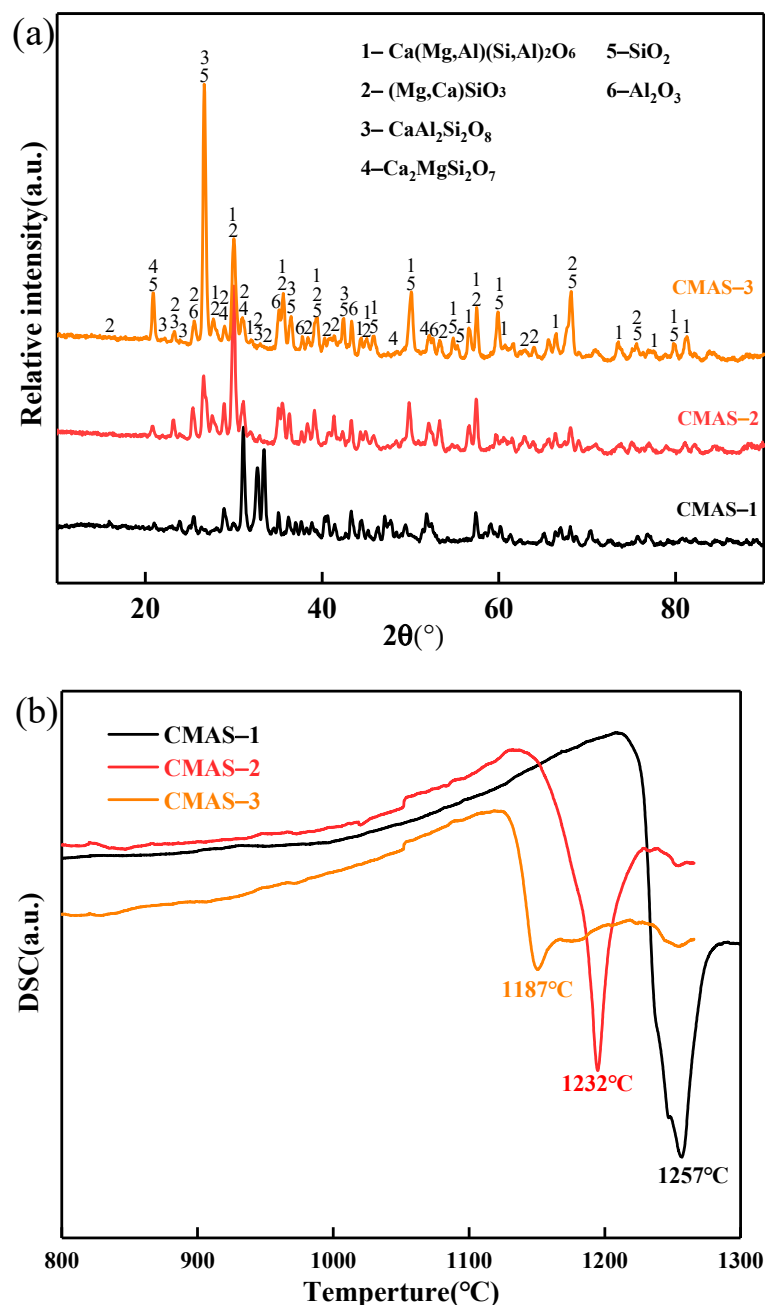


Figure 1. Three types of CMAS powders: (a) XRD patterns; (b) DSC curves.

Based on Seok et al. [10,18], Ndamka et al. introduced the basicity index (BI) of CMAS to account for its influence on melt viscosity. Here, the oxides in CMAS are categorized as alkaline (CaO , MgO), acidic (SiO_2), and amphoteric (Al_2O_3), and BI is defined as [18,19]

$$BI = \frac{\sum \text{Basic oxides (mol\%)}}{\sum \text{Acidic oxides (mol\%)}} \quad (4)$$

Research has shown that the higher the BI of CMAS melt, the lower its melt viscosity [10,18,19]. Using Equation (4), the BI of three types of CMAS powders was calculated to be 1.27, 0.72, and 0.43, respectively. This is consistent with the results in Figure 2.

3.2. Macro Images

The macro images of the YSZ coating after corrosion are shown in Figure 3. It can be seen that the residual amount of CMAS gradually decreases with the increase in tempera-

ture. Meanwhile, at the same temperature, the residual amount of CMAS gradually rises as its viscosity increases. Due to the high melting point of CMAS-1, there is a large residual amount of CMAS. With the extension of time, almost all CMAS penetrates into the interior of the YSZ coating.

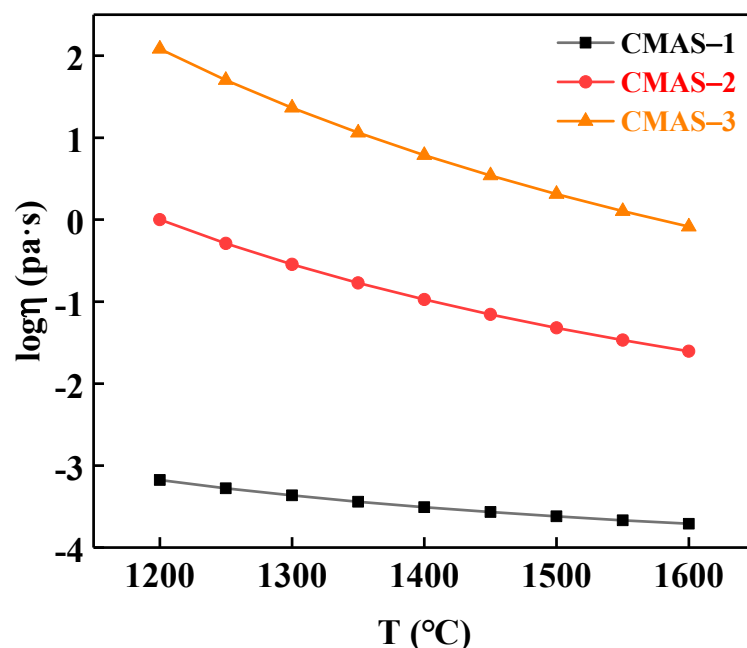


Figure 2. The viscosity results of three types of CMAS powders.

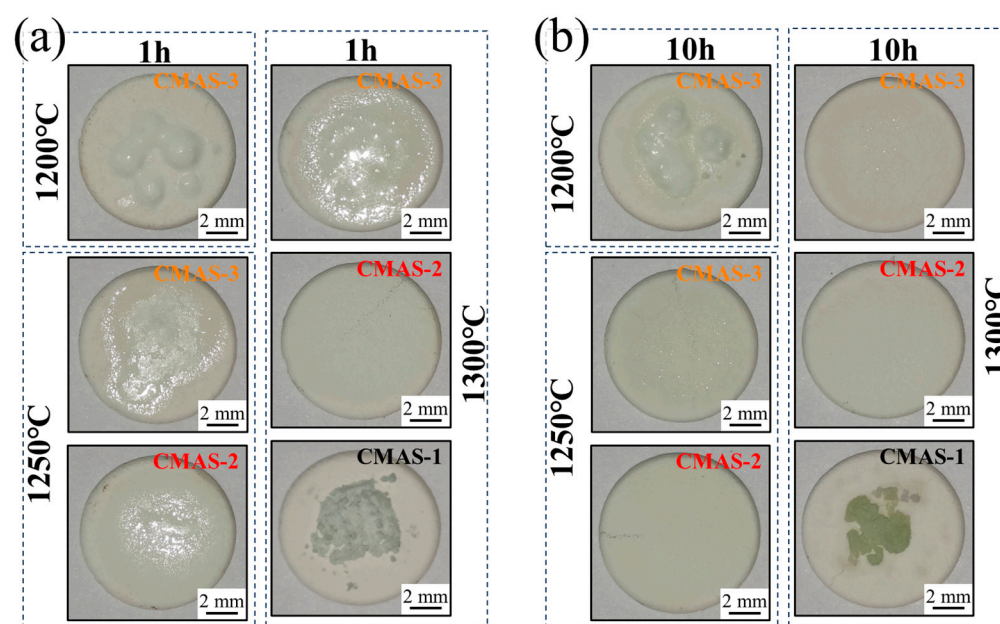


Figure 3. Macro images of the YSZ coating after corrosion at different temperatures (1200 °C, 1250 °C, and 1300 °C): (a) 1 h; (b) 10 h.

3.3. Morphology of Cross-Section

The cross-section morphology of the YSZ coating after corrosion is shown in Figure 4. It can be seen that except for CMAS-1 powder after heat treatment at 1300 °C/1 h, all other powders penetrated into the coating interior after heat treatment, filling pores and microcracks within the coating and resulting in a reduction in coating porosity.

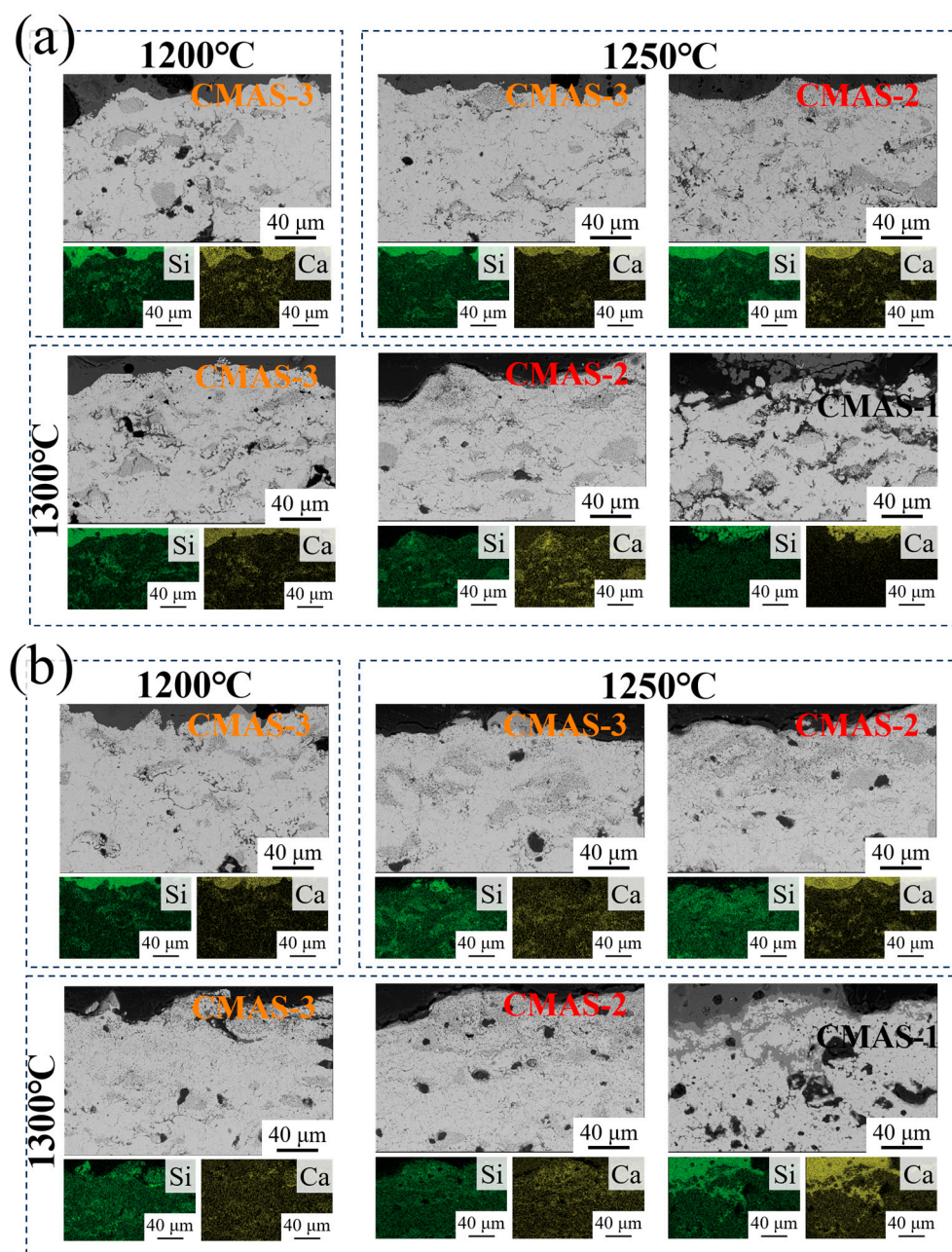


Figure 4. The cross-section morphology of the YSZ coating after CMAS corrosion at different temperatures (1200 °C, 1250 °C, and 1300 °C): (a) 1 h; (b) 10 h.

4. Discussion

CMAS infiltration impairs the thermal insulation performance of coatings. Meanwhile, the phase transformation of t' -ZrO₂ also raises the thermal conductivity of coatings [20,21]. Therefore, to reveal the influence of different CMAS compositions on the phase transformation of YSZ, the phase compositions of YSZ coating after heat treatment via Raman spectroscopy were characterized, and the results are shown in Figure 5. It can be observed that m -ZrO₂ forms in all coatings corroded by powders except those exposed to CMAS-1 powder corrosion. This may be because CMAS-1 has the highest melting point despite its lowest viscosity.

The m -ZrO₂ volume fraction can be estimated according to Equation (5) [22,23]:

$$V_m = \frac{I_m^{178} + I_m^{189}}{0.33 \cdot (I_t^{145} + I_t^{256}) + I_m^{178} + I_m^{189}} \quad (5)$$

where I_m and I_t are the peak intensity of the m-ZrO₂ and t-ZrO₂, respectively, and the superscript is the corresponding wave number.

The results in Figure 5 were calculated using Equation (5), and the results are presented in Figure 6. It can be seen from that the content of m-ZrO₂ rises gradually with the extension of holding time and the increase in temperature. Meanwhile, the content of m-ZrO₂ decreases progressively as the coating depth increases. Under all tested conditions, the content of m-ZrO₂ after CMAS-3 corrosion is higher than that after CMAS-2 corrosion.

The reactivity between molten CMAS and YSZ can be evaluated by the optical alkalinity (OB) difference based on Lewis acid–base theory. The theoretical OB values of the CMAS can be calculated by Equation (6) [8,24]:

$$\Lambda = X_1 \cdot \Lambda(\text{CaO}) + X_2 \cdot \Lambda(\text{MgO}) + X_3 \cdot \Lambda(\text{AlO}_{1.5}) + X_4 \cdot \Lambda(\text{SiO}_2) \quad (6)$$

where X_i ($i = 1, 2, 3, \dots$) stand for the oxide i mole fraction, and the corresponding Λ is its OB. The Λ values of the oxides are shown in Table 2.

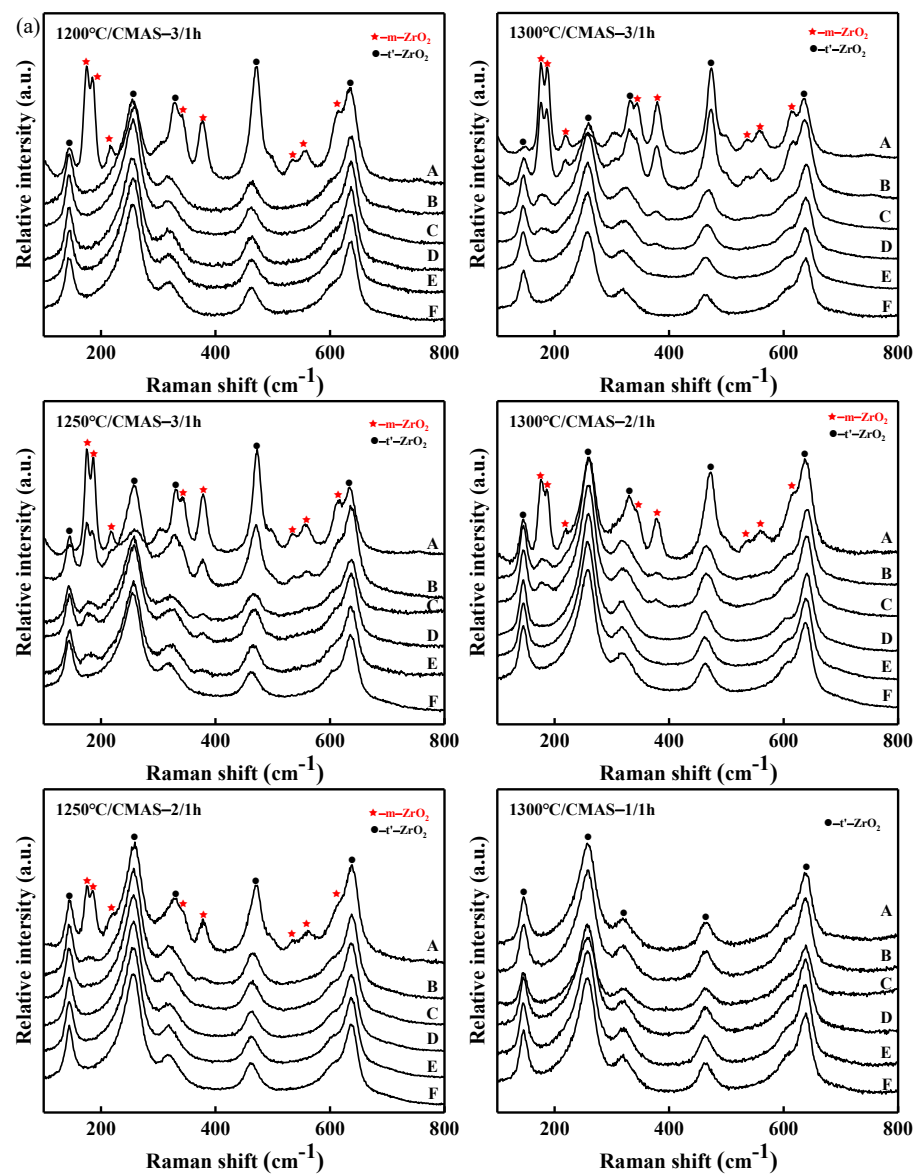


Figure 5. Cont.

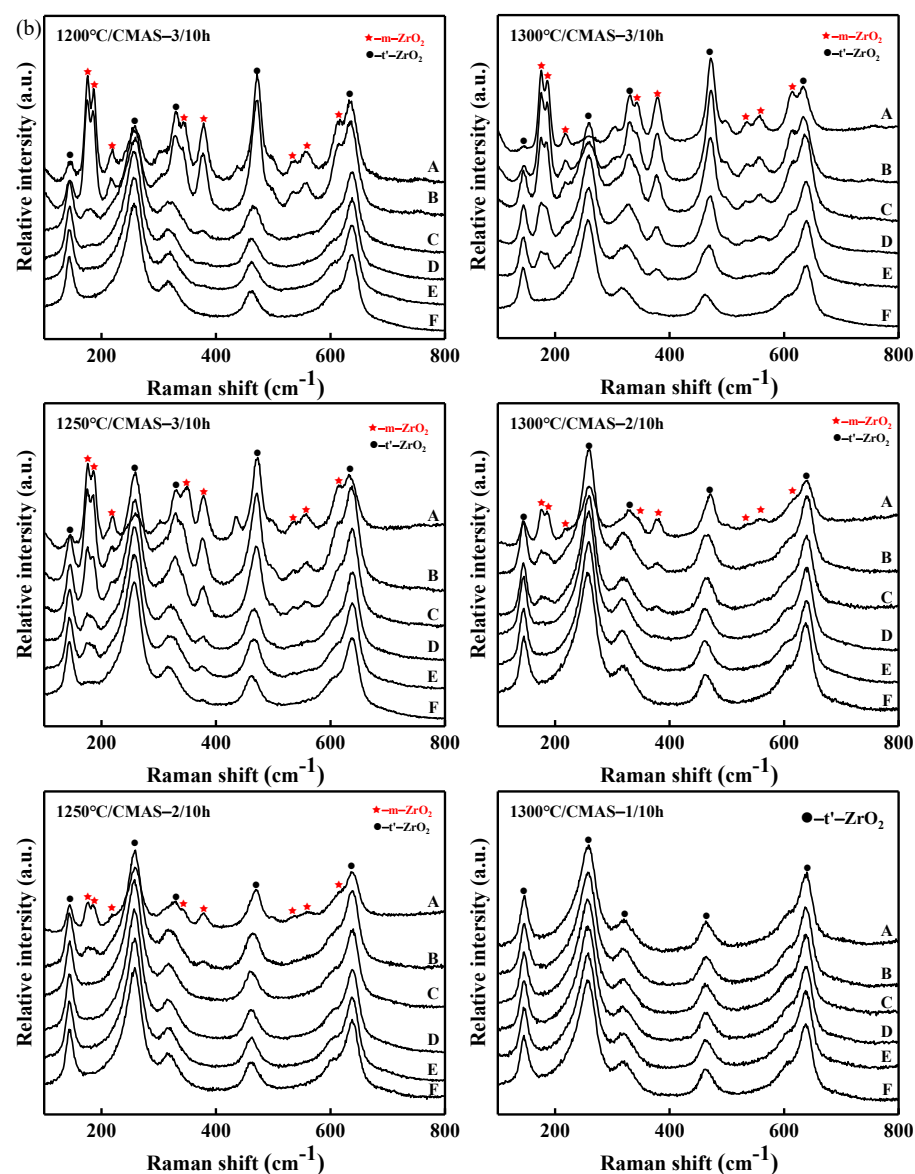


Figure 5. Raman spectra of YSZ coating cross-sections after CMAS corrosion at different temperatures (1200 °C, 1250 °C, and 1300 °C): (a) 1 h; (b) 10 h (A refers to the near-surface of the coating, and A to F each extend downward by 10 μm).

Table 2. The Λ values of the oxides [24].

Oxide	CaO	MgO	Al ₂ O ₃	SiO ₂
Λ	1.00	0.78	0.60	0.48

The Λ values in Table 2 are substituted into Equation (6) to obtain the Λ values of the three types of CMAS powders, and the difference ($\Delta\Lambda$) between YSZ and them, as shown in Table 3. It can be seen that CMAS-3 has the highest tendency to react with YSZ, while CMAS-1 has the lowest, which is consistent with the results shown in Figure 6.

Table 3. The Λ and $\Delta\Lambda$ values of the YSZ and three types of CMAS.

	YSZ	CMAS-1	CMAS-2	CMAS-3
Λ	0.8505 [8]	0.767	0.6942	0.6318
$\Delta\Lambda$	-	0.0835	0.1603	0.2187

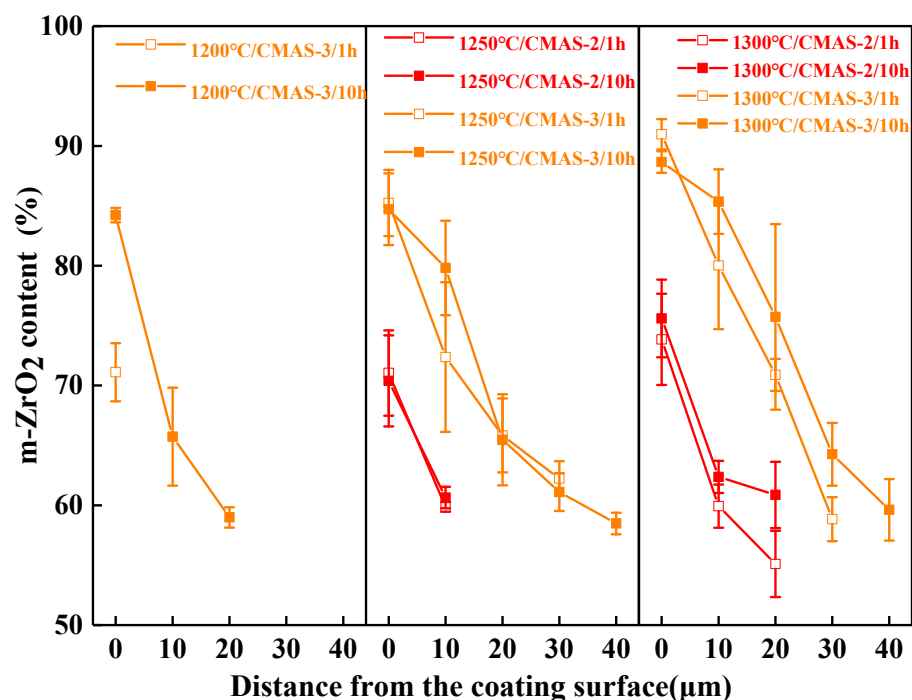


Figure 6. Depth-dependent m-ZrO₂ phase content at different positions in Figure 5.

Under capillary action, the penetration depth (L) of the CMAS melt into the coating has been found to vary with time (t) according to the Washburn equation [9,25]:

$$L = \sqrt{\frac{r\sigma_{LV}\cos\theta}{2\eta}}\sqrt{t} \quad (7)$$

where r represents the equivalent radius of the capillary; θ represents the equilibrium contact angle of the melt on the TC surface; and σ_{LV} and η are the surface tension and viscosity of the melt, respectively. According to Equation (7), within the same time (t), the penetration depth (L) is negatively correlated with the melt viscosity (η). Since CMAS-3 has the lowest melting point and the highest viscosity, it slows down the penetration rate of CMAS-3, extends its reaction time with YSZ, and generates more m-ZrO₂. The apparent contradiction that CMAS-3 exhibits the highest viscosity yet the strongest reaction tendency is resolved by distinguishing physical infiltration (controlled by viscosity) from chemical degradation (controlled by melting point and optical basicity). Although CMAS-3 has the highest viscosity, its lowest melting point provides a longer effective reaction time, thereby promoting more severe chemical attack.

Quantitative penetration depth measurements reveal the following distinct infiltration behaviors. At 1250 °C, CMAS-3 reaches $497 \pm 9 \mu\text{m}$ at 1 h (CMAS residual $161.3 \pm 8.4 \mu\text{m}$) and $913 \pm 14 \mu\text{m}$ at 10 h (without CMAS residual), while CMAS-2 reaches $815 \pm 12 \mu\text{m}$ at 1 h (CMAS residual $97.2 \pm 7.4 \mu\text{m}$) and achieves full penetration (without CMAS residual) at 10 h. This difference is consistent with the higher viscosity of CMAS-3, which retards the infiltration rate, and follows the Washburn-type relationship, where penetration depth increases with time and decreases with viscosity. At 1200 °C, CMAS-3 already penetrates to $389 \pm 9 \mu\text{m}$ at 1 h (CMAS residual $182.5 \pm 5.5 \mu\text{m}$) and $555 \pm 4 \mu\text{m}$ at 10 h (CMAS residual $151.5 \pm 4.5 \mu\text{m}$), confirming its early infiltration onset due to its lower melting point. At 1300 °C/1 h, CMAS-3 shows an average penetration of $893 \pm 32 \mu\text{m}$ (CMAS residual $81.4 \pm 12.0 \mu\text{m}$), while CMAS-2 achieves full penetration (without CMAS residual). In contrast, CMAS-1 shows no penetration at 1300 °C/1 h (CMAS residual $260 \mu\text{m}$) and only $105 \pm 6 \mu\text{m}$ at 10 h (CMAS residual $239.3 \pm 4.0 \mu\text{m}$), despite having the lowest viscosity,

confirming that its high melting point delays the initiation of infiltration. At 1300 °C/10 h, both CMAS-2 and CMAS-3 achieve full penetration (without CMAS residual), indicating that the differences in melting point and viscosity become less critical when the temperature is sufficiently high.

5. Conclusions

YSZ coating was prepared via APS, and the corrosion performance of the coating under three types of CMAS at 1200 °C, 1250 °C and 1300 °C was studied. The main conclusions are as follows:

- (1) CMAS melting point: CMAS-1 > CMAS-2 > CMAS-3; CMAS viscosity: CMAS-1 < CMAS-2 < CMAS-3; CMAS and YSZ reaction tendency: CMAS-1 < CMAS-2 < CMAS-3;
- (2) The reaction tendency between CMAS and YSZ is highly sensitive to temperature.
- (3) Based on the measured penetration depths, it is concluded that CMAS infiltration into YSZ coatings is governed by both melting point and viscosity—the melting point initiates infiltration, while the viscosity controls the penetration rate.

Therefore, when designing CMAS-resistant coatings, it is necessary to consider the impacts caused by varying CMAS compositions in different regions.

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Data Availability Statement: The data presented in this study are available within the article. Additional information supporting the findings of this work are available from the corresponding author upon reasonable request.

Conflicts of Interest: Author Yong Shang was employed by the Beihang Chengdu Aero Engine Innovation Research Institute Co. Ltd. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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