Review

Thermal barrier coatings produced by chemical vapor deposition

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Abstract

Yttria stabilized zirconia (YSZ) can be employed as thermal barrier coatings (TBCs) on Ni-based super alloys in gas turbines and aircraft engines. The YSZ coatings have been fabricated by atmospheric plasma spraying or electron-beam physical vapor deposition. The increase in operation temperature of gas turbines demands another fabrication process to obtain high quality TBCs. Chemical vapor deposition (CVD) can be an alternative route to prepare TBCs due to excellent conformal coverage and columnar microstructure. This paper reviews the fabrication of YSZ films by conventional thermal CVD and plasma CVD intended for TBCs. A new laser CVD developed by our group with a high deposition rate of 660 μm h⁻¹ was also briefly introduced.

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Keywords: Thermal barrier coating; Chemical vapor deposition; Plasma CVD; Laser CVD; Yttria stabilized zirconia; Deposition rate; Columnar structure

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1. Introduction

Since the metal components in gas turbines and aircraft engines, mainly Ni-based superalloys, will be exposed to high temperature gases over 1573 K, thermal barrier coatings (TBCs) should be used to reduce the heat transfer to the base metals. TBCs would extend the lifetime of the metal components with improving the efficiency of gas turbines and allowing higher operating temperatures. Yttria stabilized zirconia (YSZ) has been used for TBCs because of its suitable low thermal conductivity, chemical stability in combustion atmospheres and high thermal expansion coefficient. To ensure the performance of TBCs, the coatings should have requirements leading to the appropriate thermal–mechanical stability, high deposition rates and availability to coat complex shaped areas.

Two kinds of deposition techniques have been employed to produce TBCs; atmospheric plasma spraying (APS) and electron beam-physical vapor deposition (EB-PVD). The APS process typically produces TBCs with laminar structures and high porosity (10–20%) allowing a low thermal conductivity (0.5–1.0 W m⁻¹ K⁻¹). The EB-PVD process yields high deposition rates about 250 μm h⁻¹ and columnar grain structures, where individual columns would prevent the tensile stresses produced by a thermal expansion misfit between coatings and metal components. Although both techniques are widely utilized to apply TBCs on blades and combustion chambers, they are characterized by a limited capability to coat complex shaped components due to their line-of-sight nature. Therefore, an alternative process to satisfy the demands of TBCs would be of high technological impact.
Chemical vapor deposition (CVD) is a highly attractive process for TBCs because of its advantageous capability to coat complex surfaces uniformly with excellent conformal coverage [1–12]. However, deposition rates of CVD have been usually low typically below 10 $\mu$m h$^{-1}$, and then many efforts have been made to enhance the deposition rates of CVD for TBCs [13–19]. Recently, the use of auxiliary energy sources in CVD such as plasma and laser have resulted significant enhancement of deposition rates [16,18,21]. In particular, we have demonstrated that deposition rates have significantly improved to as high as 660 $\mu$m h$^{-1}$ in laser CVD [21]. YSZ coatings prepared by plasma CVD have exhibited columnar structure and non-transformable tetragonal $t'$ phase [18,20]. Therefore, CVD will have more attention in TBCs. This paper briefly reviews the CVD routes to produce TBCs and our recent results by laser CVD.

2. Thermal CVD of YSZ films

Conventional thermal-activated CVD has typically led to YSZ thin films having single cubic or mixed monoclinic and 2. Thermal CVD of YSZ films

Table 1 summarizes CVD conditions and several properties for YSZ intended for TBCs. Wahl et al. [15] reported the use of Zr(thd)$_3$ and Y(thd)$_3$ precursors in a vertical hot-wall reactor at 900–1300 K and 1 kPa. The precursor vapors were separately carried by argon gas, and reacted with preheated oxygen. Under optimized conditions, thick coatings with a columnar structure were obtained at a high deposition rate of about 50 $\mu$m h$^{-1}$. Although the composition of coatings widely varied from 1.4 to 19 mol% Y$_2$O$_3$, their crystalline phase was predominantly cubic. By increasing evaporation temperature of precursors in a gas phase, much higher deposition rates up to 100 $\mu$m h$^{-1}$ were attained. However, coatings exhibited poor adhesion to the substrates and the columnar structure tended to disappear.

Dubourdieu et al. [16] employed a solid single-source delivery system to deposit YSZ films from Y(thd)$_3$ and Zr(thd)$_4$ precursors at 723–1123 K and 0.40 kPa. The precursors mixed in the desired ratios were packed into a pyrex tube. The delivery system permitted the tube to move forward and pass trough a sharp temperature gradient between a cooled jacket and a micro-heater. In this way, the mixed precursors were instantly vaporized at temperatures between 573 and 623 K. The flux of precursor vapors was varied by controlling the speed of the moving tube. Oxygen or nitrous oxide was used as an oxidant gas. This delivery system allowed a wide range of deposition rates from 0.12 to 42 $\mu$m h$^{-1}$ with a precise control of the film deposition.

Table 1

| Process          | Precursors             | Deposition temperature (K) | Phase | Y$_2$O$_3$ content (mol%) | Deposition rate ($\mu$m h$^{-1}$) | Coating thickness ($\mu$m) | Reference |
|------------------|------------------------|---------------------------|-------|---------------------------|----------------------------------|---------------------------|-----------|
| Thermal CVD      | Zr(O-i-C$_4$H$_9$)$_4$, Y(dpm)$_3$ | 873–1173                  |       | 3–5                       | 0.2–2.5                          |                           | [1,2]     |
| Thermal CVD      | Zr(O-n-C$_4$H$_9$)$_4$, Y(thd)$_3$ | 873–1123                  |       | 5                          | 5–9                              | 1–4                       | [3]       |
| Thermal CVD      | Zr(dpm)$_3$, Y(thd)$_3$ | 773–1023                  |       | Cubic                     | >5                               | 0.3–2                     | [4]       |
| Thermal CVD      | ZrCl$_4$, YCl$_3$      |                           |       | Cubic                     | 1.5–2.0, 25                      |                           | [5,6]     |
| Thermal CVD      | Zr(thd)$_3$, Y(thd)$_3$ | 923–1123                  |       | Cubic                     | 0.7–1.2                          |                           | [7]       |
| Plasma CVD       | Zr(thd)$_3$, Y(thd)$_3$ | 773                       |       | Cubic                     | 0.3–0.8                          |                           | [8]       |
| Plasma CVD       | Zr(dpm)$_3$, Y(dpm)$_3$ | 773                       | m + c | 4–5                       | 3–6                              |                           | [9]       |
| Aerosol CVD      | Zr(acac)$_3$, Y(acac)$_3$ | 823–973                  |       | Cubic                     | >7                               | 3–6                       | [9]       |
| Aerosol CVD      | Zr(dpm)$_3$, Y(dpm)$_3$ | 773–1173                  |       | Cubic                     | >8                               | 2                         | [10]      |
| Combustion CVD   | Y 2-EH, Zr 2-EH        |                           |       |                           | 2.5–7.5                          | 1.2–5.2                   | [12]      |

dpm, Dipivaloylmethanato; acac, acetylacetonato; thd, tetrarmethyl-heptanedionato; (O-i-C$_4$H$_9$)$_4$, tertiary butoxide; (O-n-C$_4$H$_9$)$_3$, normal butoxide; 2-EH, 2-ethylhexanoate.
composition from 5 to 30 mol% \( \text{Y}_2\text{O}_3 \). YSZ films exhibited highly aligned columnar or fine-grained microstructures. Typical film thickness was varied from about 0.2 to 3 \( \mu \text{m} \).

YSZ films showed either mixed cubic and monoclinic or pure cubic phases mainly depending on the substrate temperature, \( \text{Y}_2\text{O}_3 \) content and film thickness. We have prepared YSZ films intended for TBCs with high deposition rates [17]. Fig. 1 shows YSZ films prepared from \( \text{Zr(dpm)}_4 \) and \( \text{Y(dpm)}_3 \) precursors at 1073 K and 0.8 kPa for 0.9 ks. The precursors were separately vaporized at 593 and 433 K, respectively, and carried by argon gas. Oxygen was added to precursor vapors in a mixing zone above the substrate. Well-developed columnar grains were observed in the fractured cross-sections of YSZ films prepared on quartz substrates as shown in Fig. 1(a). Since the Hastelloy-XR substrate is ductile and cannot be fractured, a polished cross-section of YSZ film was demonstrated in Fig. 1(b). The columnar grains were identified and adhered well to the alloy substrate. Crystal structure of coatings fitted to a tetragonal phase with a significant (200) orientation. YSZ lattice parameters were determined as \( a = 0.5101 \text{ nm} \) and \( c = 0.5168 \text{ nm} \). According to the relationship between lattice parameters and \( \text{Y}_2\text{O}_3 \) fraction [22], \( \text{Y}_2\text{O}_3 \) content in the coating was estimated to be about 3.0 mol% \( \text{Y}_2\text{O}_3 \), which was consistent with that obtained by EPMA analysis (2.7 mol% \( \text{Y}_2\text{O}_3 \)). Fig. 2 summarized the temperature dependence of deposition rates for YSZ films prepared by conventional thermal CVD reported in literatures [15,17,24–26]. The activation energy of deposition rates in a low temperature region is 100–132 kJ mol\(^{-1}\) suggesting a chemical reaction limited process [24–26]. The deposition rates become less dependent on temperature with increasing temperature. The activation energy at 900–1100 K is 60 kJ mol\(^{-1}\) [17]. This smaller value implies that the rate-controlling step could be a diffusion process in a gas phase. It is generally understood that the chemical reactions are fast enough at high temperatures and the deposition rates are controlled by mass transfer (diffusion) in a gas phase [27]. Therefore, the diffusion-control region has been commonly selected to obtain high deposition rates in CVD [28]. The deposition rates are also strongly dependent on the heating method of substrate. CVD can be divided into two types according to a heating method of substrate. The one is a hot-wall type where the substrate is indirectly heated by the hot walls of CVD chamber, and the others is a cold-wall type where the substrate is directly heated with keeping the walls of CVD chamber cold. Since the deposition reactions in the hot-wall type CVD easily occur in a gas phase and at the hot-wall surface as well, the deposition rate and efficiency are usually low. In the cold-wall type CVD, the deposition reactions are restricted at around the substrate and high deposition rates can be attained [29]. We have constructed a cold-wall type CVD chamber, and optimized the deposition conditions and the geometric configuration of chamber to maximize the deposition rates. Then, we have achieved a high deposition

Table 2

| Process       | Precursors          | Deposition temperature (K) | Phase                      | \( \text{Y}_2\text{O}_3 \) content (mol%) | Deposition rate (\( \mu \text{m} \text{h}^{-1} \)) | Coating thickness (\( \mu \text{m} \)) | Reference |
|---------------|---------------------|----------------------------|----------------------------|------------------------------------------|---------------------------------------------|--------------------------------------|-----------|
| Thermal CVD   | \( \text{Zr(thd)}_4, \text{Y(thd)}_3 \) | 900–1300                   | Cubic                      | 1.4–19                                   | 50                                          |                                      | [15]      |
| Thermal CVD   | \( \text{Zr(thd)}_4, \text{Y(thd)}_3 \) | 723–1123                   | Cubic + monoclinic         | 5–30                                    | 0.12–42                                   | 1                                    | [16]      |
| Thermal CVD   | \( \text{Zr(dpm)}_4, \text{Y(dpm)}_3 \) | 873–1173                   | Tetragonal                 | 2.7                                    | 100                                        | 25                                   | [17]      |
| Plasma CVD    | \( \text{ZrCl}_3, \text{Y(thd)}_3 \) | 973–1173                   | \( t \)                    | 3–4.4                                   | 100–250                                   | 65–200                              | [18,19]  |
| Laser CVD     | \( \text{Zr(dpm)}_4, \text{Y(dpm)}_3 \) |                           |                           | \( >300 \)                              | 100                                        |                                      | [21]      |

\( t \), Non-transformable tetragonal phase; thd, tetramethyl-heptanedionate; dpm, dipivaloylmethanato.

![Fig. 1. YSZ coating prepared by thermal-activated CVD on (a) fused quartz (fractured cross-section) and (b) Hastelloy substrates (polished cross-section) [17].](image)
efficiency of 25–35% and the highest deposition rate of 108 \( \mu m h^{-1} \) by thermal CVD [17]. This value is higher than typical yields in conventional thermal-activated CVD (10–15%) [15], and comparable to that obtained by solid precursor powder delivery system (25%) [16].

### 3. Plasma CVD of YSZ coatings

An alternative route to further increase in deposition rates of YSZ coatings has been investigated by selecting an auxiliary energy source to enhance chemical reactions in CVD. The potentiality of microwave plasma-enhanced CVD (PE-CVD) to yield ZrO\(_2\) coatings at high deposition rates for TBCs was first demonstrated by Bertrand and Mevrel [13]. Columnar ZrO\(_2\) coatings with high deposition rates about 120–150 \( \mu m h^{-1} \) were obtained at 573–1073 K using \( ZrCl_4 \) in a microwave plasma-enhanced CVD reactor. Preauchat and Drawing [18] has demonstrated the ability of plasma-enhanced CVD to produce columnar 65–200 \( \mu m \)-thick YSZ coating with high deposition rates from 100 to more than 250 \( \mu m h^{-1} \). YSZ coatings exhibited the desirable non-equilibrium tetragonal t\(_0\) phase with a strong (200) orientation. The PE-CVD coatings were prepared at 973 or 1173 K and 106.4 Pa using a microwave power about 1700 W. \( ZrCl_4 \) and \( Y(thd)_3 \) precursors were separately evaporated at 443–503 K, where \( Y_2O_3 \) contents varied from 3 to 4.3 mol%. Coatings obtained at 973 K consisted of fine and irregular aligned columns while those prepared at 1173 K exhibited well-developed columns. The morphology of the columns was as dense cores with feather-like flanks similar to that observed in EB-PVD coatings. Fig. 3 shows the columnar structure of a YSZ coating prepared by plasma-enhanced CVD process.

The high-temperature stability of PE-CVD coatings was investigated by isothermal-annealing at 1373–1673 K and thermal-cycling at 1373 K [20]. The PE-CVD coatings exhibited a good resistance to thermal cycling up to 400 cycles with a lifetime similar to that of EB-PVD coatings. The sintering of columns was more significant in the more porous coatings prepared at 973 K. The coatings prepared at 1173 K showed similar thermal conductivity (1.6–1.7 W m\(^{-1}\) K\(^{-1}\) at 1373 K) as those obtained by EV-PVD (1.5–1.8 W m\(^{-1}\) K\(^{-1}\) at 1373 K).

### 4. Laser CVD of YSZ coatings

We have developed a high power laser assisted CVD process to prepare YSZ coatings. The laser CVD process has enabled high deposition rates up to 660 \( \mu m h^{-1} \) and desirable columnar structural for TBCs. The YSZ coatings were prepared in a specially designed vertical and cold-wall CVD apparatus [21]. An Nd:YAG laser beam (wavelength: 1063 nm) operating up to 260 W was emitted through a quartz window onto substrates placed on a heated stage. A large un-focused laser spot (about 15 mm in diameter) allowed uniform coating of substrates (13 mm \( \times \) 13 mm \( \times \) 2 mm). The temperature of the substrate stage was varied from room temperature to 1023 K during deposition. The precursors, \( Zr(dpm)_4 \) and \( Y(dpm)_3 \), were kept at 493 and 443 K, respectively. The vapors were carried by argon gas to the reactor chamber through a double tube nozzle perpendicular to the substrate. Oxygen gas was separately
introduced into the chamber 15 mm above the substrate. Total pressure was controlled at 0.93 kPa.

The laser power and the temperature of the substrate stage have a strong influence on deposition rate, crystalline structure and morphology of YSZ coatings. In a low laser power (less than about 60 W), low deposition rates about 1–3 μm h⁻¹ were observed. An increase in laser power has led to a sharp rise in deposition rates. Thus in higher laser powers, more than 100 W, deposition rates reached almost constant values about 300 μm h⁻¹. A further increase of precursor concentration in the gas phase has resulted in high deposition rates to 660 μm h⁻¹. A high laser power combined with a high precursor concentration has caused particular plasma formation associating significantly high deposition rates. Fig. 4 shows a plasma emission spectrum at a laser power of 250 W and a deposition rate of 230 μm h⁻¹ [30]. The spectrum showed a broad peak, and no sharp peak characteristic to specific dissociation process in usual low temperature plasma was identified. The black body emission, i.e. the plank distribution spectrum, is included in Fig. 4. The experimental spectrum was similar to but slightly shaper than that of the plank distribution. The precursor vapors might dissociate by the high power laser forming the plasma and reactive clusters. Such clusters activated by the laser radiation could yield the high deposition rate and efficiency.

The influence of the stage temperature on morphology of YSZ coatings is shown in Fig. 5. The laser power was kept constant at 250 W. The cross-sectional SEM images revealed a pattern of columnar growth. As the stage temperature increased from 373 to 1023 K, the columns have developed well-grown features. Columns with broad and rough endings have been associated to low temperatures. Then, columns become round and tall with occasional faceted tips. At the highest stage temperature, columns turn out to be well-developed with typical sharp and faceted tips similar to EB-PVD coatings [31].

Fig. 4. A plasma emission spectrum at a laser power of 250 W [30].

Fig. 5. YSZ coatings prepared by laser CVD at a laser power of 250 W and substrate stage temperatures of: (a) 1023 K; (b) 623 K and (c) 373 K.
5. Conclusions

TBCs have been industrially produced by APS and EB-PVD on complicated shaped components of gas turbines and aircraft engines. CVD could be a potential alternative process because of its conformal coverage capability and desirable morphology. Several routes have been investigated to apply CVD for TBCs mainly to enhance the deposition rates. The sufficient results have been achieved by using auxiliary energy sources in CVD of YSZ such as plasma and laser. The present status indicates that CVD is a process able to produce YSZ coatings with columnar structures at deposition rates higher than 300 $\mu$m h$^{-1}$ in a laboratory scale. The newly developed laser CVD could be particularly attractive to obtain high deposition rates as high as 660 $\mu$m h$^{-1}$. CVD process would contribute to develop both processing and performance of TBCs.

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