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Epitaxial growth during the rapid solidification of plasma-sprayed molten TiO₂ splatShu-Wei Yao, Tao Liu, Chang-Jiu Li^{*}, Guan-Jun Yang, Cheng-Xin Li

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ABSTRACT

In this study, epitaxial growth during the rapid solidification of plasma-sprayed molten TiO₂ droplet was studied. The crystallographic structure of the TiO₂ splats deposited on rutile and α -Al₂O₃ substrates at 150, 300 and 500 °C was characterized by high resolution transmission electron microscopy and electron back scattering diffraction. The results reveal that homo-epitaxial and hetero-epitaxial TiO₂ splats can be formed at the deposition temperature of 500 °C. Crystal orientation is another key factor influencing the epitaxial growth process. It is easier to form an epitaxial TiO₂ splat with the (001) orientation in the crystal growth direction. Based on the experimental results, a competition mechanism between heterogeneous nucleation and epitaxial growth was proposed to understand the phenomena of epitaxy during the rapid solidification process. The effect of undercooling degree, crystal orientation and deposition temperature on the epitaxial growth of TiO₂ splat was examined. The simulation results are in close agreement with the experimental observations.

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1. Introduction

Plasma-sprayed coatings are built up through the successive deposition of individual fully molten or/and semi-molten particles [1]. Following the high-speed impact, molten droplets rapidly spread and solidify into thin splats. Generally, plasma-sprayed splats present a regular disc shape with a thickness of 0.5–3 μ m, when they are deposited on a flat surface at a temperature above the transition temperature [2,3]. The transition temperature ranges from 150 to 300 °C depending on the combination of splat and substrate materials [3]. During plasma spraying, the mean cooling rate of ceramic splats is about 10⁴–10⁵ K/s [4]; for metal splat, the mean cooling rate is up to 10⁶–10⁸ K/s. However, the maximum cooling rate would be much higher than the average, especially at the early stage of cooling process. Owing to the rapid cooling and solidification, plasma-sprayed splats and coatings always exhibit metastable phases or even amorphous phase [5–11]. In addition, the polycrystalline splats contain fine elongated grains, with the longitudinal axis perpendicular to the substrate surface [9,10,12–15]. Therefore, it was regarded as impossible to deposit

epitaxial splats or coatings by plasma spraying.

Generally, plasma-sprayed ceramic coatings exhibit a typical lamellar structure with numerous non-bonded interface [16,17]. The individual ceramic splats always exhibit a columnar grain structure. Gray et al. [18] prepared a continuous columnar structured YSZ coating at higher deposition temperature. Observing from the transmission electron microscope (TEM) image, Sampath et al. [19] found a columnar grain extending from one splat into an adjacent splat in the continuous-columnar-structured YSZ coatings. However, there also exist non-epitaxial grains and non-bonded lamellar interface just beside the epitaxial grain. Thus, both epitaxial and non-epitaxial grains can be formed in the continuous-columnar-structured coatings. Moreover, Chraska et al. [13] occasionally observed epitaxial columnar grains in the subsequent splat, when two YSZ splats were successively deposited on a stainless steel substrate at about 450 °C. Recently, Yang et al. [9,20] investigated the effect of deposition temperature on the crystal structure of plasma-sprayed Al₂O₃ and 8YSZ splats. They found that epitaxial Al₂O₃ splats can be formed on α -Al₂O₃ substrate at 900 °C. In addition, epitaxial growth occurs in the 8YSZ splats deposited on the cubic 8YSZ (8 mol % Y₂O₃-ZrO₂) and the tetragonal 3YSZ substrates at 900 and 1200 °C, respectively. This fact implies that the epitaxial growth of plasma-sprayed molten splat depends not only on the deposition temperature, but also on the substrate crystalline structure.

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It is a very interesting phenomenon that epitaxial growth occurs during the rapid solidification process. In addition, epitaxial growth is conducive to the design of coating microstructure, as well as the improvement of coating properties and performance. For example, the formation of coherent or semi-coherent interface due to epitaxy can significantly improve the interlamellar bonding strength and the electrical conductivity. However, epitaxial growth during plasma spraying has not been widely investigated. Gray et al. [18] considered that the deposit surface can be locally melted by a subsequent molten particle at a high deposition temperature, and then columnar directional solidification takes place in the incoming particle from the grains of the adjacent underlying layer. Wei et al. [21] and Zhang et al. [22] suggested that epitaxial growth might be possible when substrate melting occurs. However, as revealed by Li et al. [23] and Zhang et al. [24], substrate melting would not happen unless the melting point of sprayed material is much higher than that of substrate material. Thus, the epitaxial growth of plasma-sprayed ceramic splats is not attributed to local melting. On the other hand, epitaxial growth would not happen even when the substrate is locally melted by a molten particle with higher melting point, since there exist many factors influencing epitaxial growth, such as lattice mismatch. For example, Houben et al. [25] revealed that the steel substrate was locally melted due to the impact of Mo droplet. However, in this case, the substrate melting contributes to form an intermetallic layer instead of an epitaxial Mo splat. Thus, the mechanism of epitaxial growth during the rapid solidification process, such as plasma spraying, needs to be further discussed.

Titanium dioxide (TiO_2) is one of the most widely investigated materials owing to its excellent physical and chemical properties [26–29]. Although polycrystalline and amorphous TiO_2 films have been widely used in functional devices [29], the epitaxial TiO_2 film still attracts much interest due to its superior properties [26,30–32]. Up to now, high quality hetero-epitaxial and homo-epitaxial TiO_2 films have been successfully prepared by various processes [26,27,30,33–40], such as metal-organic chemical vapor deposition (MOCVD) and pulsed laser deposition (PLD). Chang et al. [33] and Chen et al. [34] obtained single-crystal rutile TiO_2 films on $\alpha\text{-Al}_2\text{O}_3$ substrates with (112 0), (0001) and (110 2) surfaces at a growth temperature of 800 °C by MOVCD. Via PLD process, Apgar et al. [40] succeeded in the growth of epitaxial anatase films on a wide range of monocrystalline perovskite substrates, which have a lattice mismatch ranging from –2.0% to 6.0% with the anatase phase. Thus, the epitaxial TiO_2 films with rutile or anatase phase can be successfully produced by controlling the substrate material. However, it is worth mentioning that, although high-quality epitaxial TiO_2 films can be successfully fabricated through the above-mentioned processes, the expensive costs of processes [41], low growth rate of film [27,39] and special requirement for film deposition [34,37] restrict their wide application in various fields.

Due to the excellent properties, thermal-sprayed TiO_2 coatings have been widely used [41,42]. Under the conventional deposition conditions, TiO_2 coatings exhibit both anatase and rutile phases with a nanocrystal structure. In this study, TiO_2 was taken for example to clearly clarify the conditions and mechanisms of epitaxial growth during the rapid solidification process. TiO_2 has the lower melting point than Al_2O_3 , YSZ and many other ceramic materials. As revealed in our previous study, the splat with a lower melting point can be well bonded to the substrate with the same material at a lower deposition temperature [43]. Thus, it was supposed that epitaxial growth would take place at a low deposition temperature for TiO_2 splat, which makes it simple to systematically investigate the epitaxial growth. Moreover, although TiO_2 has several polymorphs, the TiO_2 splat deposited on the rutile TiO_2 substrate at a temperature of 150 °C exhibits stable rutile phase [43]. Thus, there is no need to consider the phase selection issue

during the solidification of molten TiO_2 splat. In addition, since the epitaxial TiO_2 film exhibits excellent properties and has been widely studied [26,30–32], the investigation of epitaxial TiO_2 splat is conducive to broaden the application of epitaxial TiO_2 coating and the plasma spray process.

In this study, single TiO_2 splats were deposited on rutile TiO_2 and $\alpha\text{-Al}_2\text{O}_3$ substrates, and characterized by TEM and electron back scattered diffraction (EBSD). The effects of deposition temperature and crystal orientation on the epitaxial growth of TiO_2 splat were revealed. Moreover, the mechanism of epitaxial growth during rapid solidification was theoretically discussed based on the nucleation and crystal growth theories.

2. Experimental

2.1. Materials

In this study, the fuse-crushed TiO_2 powder (Sulzer-Metco, USA) with a particle size range from 10 to 45 μm was used to deposit single splats on the (110) and (001) surfaces of rutile TiO_2 , polycrystalline rutile TiO_2 substrate, the (101 0) and (110 2) surfaces of $\alpha\text{-Al}_2\text{O}_3$, respectively. All the substrates employed in this study were mirror-polished.

2.2. Splat deposition

A commercial plasma spray system in the 80 kW class (Jiujiang, China) was utilized to deposit TiO_2 splat. The spray parameters employed to fully melt TiO_2 particles were displayed in Table 1. The deposition temperatures of TiO_2 splats were controlled precisely by a copper heating stage [43]. In order to avoid splashing during the deposition of TiO_2 splat at 150 °C, the substrates were first preheated to 250 °C and then cooled down to the preset temperature. As revealed in our previous study, regular disc-shaped TiO_2 splats can be formed under these conditions [43].

2.3. Characterization of splat microstructure

The microstructure of TiO_2 splats was characterized by EBSD (Oxford, UK). The EBSD measurement was performed on splat surface at the acceleration voltage of 15 kV. In addition, selected splat samples were carefully prepared utilizing the focus ion beam (FIB, TESCAN, CZE) technique and examined using a high resolution TEM (JEOL Ltd, Japan), in order to clearly reveal the crystalline structure of splat.

3. Results

3.1. Effect of deposition temperature on the homo-epitaxial growth of TiO_2 splats

Epitaxial growth is a special crystallization process in which gaseous or liquid atoms condense on the template created by

Table 1
Spray conditions for TiO_2 splats.

Parameters	Values
Arc power (kW)	36
Arc current (A)	520
Arc voltage (V)	70
Primary plasma gas (Ar/slpm)	45
Secondary plasma gas (H_2/slpm)	4.5
Powder feed gas (N_2/slpm)	3
Spray distance (mm)	80
Traverse speed of torch (mm s^{-1})	1200
Deposition temperature (°C)	150, 300, 500

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