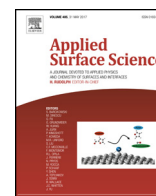




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Microstructure and properties of Al_2O_3 - ZrO_2 composite coatings prepared by air plasma spraying

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ABSTRACT

Three kinds of Al_2O_3 - ZrO_2 composite coatings with different composition were prepared by air plasma spraying on titanium alloy substrates. The compositions of the coatings were Al_2O_3 -35wt% ZrO_2 , Al_2O_3 -40wt% ZrO_2 and Al_2O_3 -45wt% ZrO_2 . The microstructure, microhardness and toughness of the three Al_2O_3 - ZrO_2 composite coatings were characterized by X-ray diffraction, scanning electron microscope, energy dispersive spectrometer and micro-Vickers. The result showed that α - Al_2O_3 transformed to γ - Al_2O_3 during the process of plasma spraying Al_2O_3 - ZrO_2 composite powders. Meanwhile, amorphous phase was formed in the Al_2O_3 - ZrO_2 composite coatings due to the rapid cooling and solidification of plasma spraying and the proper composition. There was more amorphous phase in the Al_2O_3 -40wt% ZrO_2 coating compared with the other two composite coatings. The reason was that the composition of the Al_2O_3 -40wt% ZrO_2 composite coating was closer to the eutectic point of Al_2O_3 - ZrO_2 pseudo-binary system. The Al_2O_3 -40wt% ZrO_2 composite coating with more amorphous phase had denser microstructure and lower porosity. With increasing of the content of ZrO_2 in the Al_2O_3 - ZrO_2 composite coatings, the microhardness of the coatings decreased. And the microhardness of the Al_2O_3 - ZrO_2 composite coatings was significantly higher than that of single phase Al_2O_3 coating and single phase ZrO_2 coating. Vicker indentation results showed that the Al_2O_3 - ZrO_2 composite coatings had high toughness.

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1. Introduction

Al_2O_3 is a well-known oxide material which has diverse applications in engineering ceramics [1,2]. Sintered Al_2O_3 ceramics are useful as electrical insulators, refractories, etc., and Al_2O_3 coatings on metal substrates provide improved wear and corrosion resistance [3]. However, Al_2O_3 coating is not useful for high temperature applications due to its high thermal conductivity and poor shock resistance. For thermal barrier coatings which are used to enhance the life time of parts exposed to high temperature service, the suitability of plasma-sprayed ZrO_2 on metal substrates is well documented [4–7], which is due to the good thermal insulation and shock resistance characteristics of ZrO_2 [8,9]. However, ZrO_2 coat-

ing is incompetent for wear and impact applications due to its low hardness.

Al_2O_3 - ZrO_2 composite is considered to be an interesting potential alternative to the present Al_2O_3 coating and ZrO_2 thermal barrier coating [10–17]. It is shown by the equilibrium phase diagram of the Al_2O_3 - ZrO_2 system [15] that there are phase transformation of ZrO_2 and eutectic transformation of Al_2O_3 - ZrO_2 during the heating and cooling process, in addition, Al_2O_3 and ZrO_2 have a complete miscibility in liquid state, which are beneficial to increase the density of the Al_2O_3 - ZrO_2 composite coating.

In thermal spraying process, such as air plasma spraying, the slow cooling condition to reach equilibrium is unfeasible. Coatings are not expected to show the same structure as predicted by equilibrium phase diagram, because of the rapid solidification and non-equilibrium cooling rate during the spraying process. Conversely, some metastable structures might be formed in the composite coatings.

The objective of this study was to investigate the influence of composition of feedstock powders on the microstructure,

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Table 1
The main operating parameters for plasma spraying.

	Voltage (V)	Current (A)	Primary gas (Ar) flow rate (L min ⁻¹)	Secondary gas (H ₂) flow rate (L min ⁻¹)	Spray distance (mm)
Ni-Al bond coating	65	500	70	20	~100
the composite coating	70	500	80	20	~100

microhardness and toughness of Al₂O₃-ZrO₂ composite coatings preparing by air plasma spraying on titanium alloy substrates. In addition, the phase evolution and microstructure evolution of the Al₂O₃-ZrO₂ composite powders during air plasma spraying, the influence of composition on the formation of amorphous phase, and the forming mechanism of the Al₂O₃-ZrO₂ composite coatings were investigated.

2. Material and methods

In this investigation, three kinds of Al₂O₃-ZrO₂ composite powders with different composition were used to prepare Al₂O₃-ZrO₂ composite coatings for investigating the influence of composite powders' composition on the microstructure and properties of plasma sprayed Al₂O₃-ZrO₂ coatings. The mass ratio of Al₂O₃ and ZrO₂ in the three kinds of Al₂O₃-ZrO₂ composite powders were 65:35, 60:40 and 55:45. The three kinds of Al₂O₃-ZrO₂ composite powders were labeled as AZ35 powder, AZ40 powder and AZ45 powder, respectively. For the preparing process of the composite powders, as-received powders were Al₂O₃ (α -Al₂O₃, average grain size 80 nm) and ZrO₂ (t-ZrO₂, average grain size 40 nm). These powders were blended uniformly to produce a powder mixture by wet ball-milling. The mixed powder slurries were then spray dried to form composite powders.

The titanium alloy (TC4) coupons were used as substrate samples, which were grit blasted prior to coating deposition. A bond coating of Ni/Al with thickness about 50–100 μ m was deposited onto the substrates. The three kinds of composite powders were then plasma sprayed for about 300 μ m in thickness, respectively. The GDP-2 type 50 kW plasma spraying system (Jiu Jiang Spraying Device Company, China) was employed for plasma spray processing. In this study, the main operating parameters for preparing Al₂O₃-ZrO₂ composite coatings were shown in Table 1.

The phase constitution of the powders and coatings was characterized by X-ray diffraction (XRD, D/max- γ B, Japan) with Cu K α radiation. The microstructure of the powders and coatings was characterized by scanning electron microscope (SEM, HITACHI-S4800) equipped with energy dispersive spectroscopy (EDS). Cross-sections of the as-prepared coatings were ground and polished for SEM analysis.

The porosity of the coatings was evaluated by image analysis method. The microhardness of the coatings was determined by micro-hardness tester under a normal load of 0.1 kg with a dwell time of 15 s. The toughness the coating was determined by indenting the polished coating surface with a Vickers indenter under a 0.5 kg load.

3. Results and discussion

3.1. Phase evolution of the Al₂O₃-ZrO₂ composite powders during plasma spraying

Fig. 1 shows the XRD pattern of the Al₂O₃-ZrO₂ (AZ40) composite powder. It can be seen that the composite powder consisted of α -Al₂O₃ and t-ZrO₂. Fig. 2 presents the XRD patterns of the Al₂O₃-ZrO₂ composite coatings with different compositions deposited by plasma spraying. Fig. 2a displays that the AZ35 coating consisted of

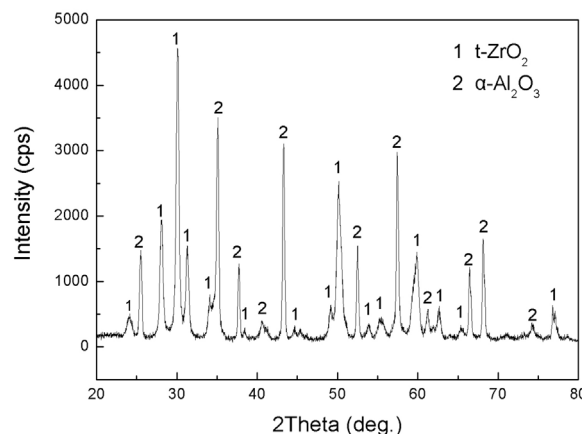


Fig. 1. XRD pattern of the Al₂O₃-ZrO₂ (AZ40) composite powder.

t-ZrO₂, γ -Al₂O₃, α -Al₂O₃ and amorphous phase. Fig. 2b shows that the AZ40 coating had t-ZrO₂ and amorphous phase. Fig. 2c exhibits that the AZ45 coating consisted of t-ZrO₂, γ -Al₂O₃, α -Al₂O₃ and amorphous phase. In comparison of Fig. 2a–c, there were more crystal phases of γ -Al₂O₃ and α -Al₂O₃ in the AZ35 coating than that of AZ45 coating. However, there was almost no Al₂O₃ crystal phase in the AZ40 coating. The reason for more Al₂O₃ crystal phase is that there was more Al₂O₃ in the AZ35 composite powder than that of the AZ45 composite powder, and therefore, the AZ35 coating had more Al₂O₃ crystal phase. Comparing Fig. 1 with Fig. 2, it can be seen that there was no phase transformation of ZrO₂ after plasma spraying. The phase transformation of Al₂O₃ took place from α -Al₂O₃ to γ -Al₂O₃. The cause for the phase transformation of Al₂O₃ during the plasma spraying process can be inferred as follow. When the composite powder entered into the plasma jet, α -Al₂O₃ was melted. Then, the melt was deposited on the surface of the metal substrate to form splat. When the temperature of the splat dropped below 1740 °C, Al₂O₃ crystal started to form. The critical nucleation free energy of α -Al₂O₃ is higher than that of γ -Al₂O₃, which means γ -Al₂O₃ is more likely to form nucleation [18], and the existence of γ -Al₂O₃ testifies it. Al₂O₃ phase in the coatings was metastable given rapid cooling and rapid solidification of plasma spraying. Meanwhile, the diffraction peaks of α -Al₂O₃ for the AZ35 coating and the AZ45 coating are weaker (Fig. 2). It indicates that Al₂O₃ phase had a good melting state in plasma jet during the plasma spraying process of Al₂O₃-ZrO₂ composite powders. And the Al₂O₃ crystal phase was mainly γ -Al₂O₃ considering the rapid cooling and solidification. Nonetheless, there was no phase transformation of ZrO₂ happened in the plasma spraying process of Al₂O₃-ZrO₂ composite powders.

There are obvious broad peaks in the XRD patterns of three Al₂O₃-ZrO₂ coatings (Fig. 2). This means amorphous existed in the three composite coatings. Fig. 2b shows that almost no crystal Al₂O₃ appeared in the AZ40 coating. Therefore, it could be inferred that Al₂O₃ crystal in the AZ40 composite powder had nearly all transformed into amorphous phase after plasma spraying. The formation causes of the amorphous phase were due to rapid cooling and rapid solidification. There was not enough time for the melted ceramic to form nucleation and grow up after quenching on the surface

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