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ARTICLE

Preparation and Properties of Y_2O_3-PF Alternating Coating on Polymer Matrix Composite Material Surface

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Abstract: High-performance yttrium oxide-phenolic resin (Y_2O_3-PF) alternating coating was prepared on epoxy resin-based composite material using supersonic plasma spraying and dual-channel powder feeding technique. Y_2O_3-coated PF (Y_2O_3/PF) powder was firstly sprayed onto the substrate, forming a transition layer, and then the spherical Y_2O_3 powder and Y_2O_3/PF powder were alternately deposited to form the composite alternating coating. Results show that the alternating coating is mainly composed of deposited Y_2O_3/PF powder. The bonding strength between coating and substrate is as high as 26.48 MPa with the single-test maximum bonding strength of 28.10 MPa, and shear strength reaches 24.30 MPa. Additionally, the heat transfer effect caused by external Y_2O_3 particles gradually softens and even melts PF, thus effectively avoiding the damage of high temperature to molecular structure and thereby promoting the crosslinking and curing effects of resin during the deposition process. In the meantime, the unmelted Y_2O_3 powder results in the shot peening effect, which washes out and eliminates the powder particles with inferior deposition effect, ultimately improving the physical and chemical properties of the alternating coating.

Key words: Y_2O_3-PF alternating coating; epoxy resin composite material; supersonic plasma spraying; mechanical property

Polymer-based composites (PMCs) are widely used in the aviation, aerospace, automotive, and architecture fields due to their high strength, low density, excellent heat resistance, and fine corrosion resistance[1–4]. By preparing functional coatings on PMCs, the damage resistance, mechanical property, and heat resistance are significantly improved, thereby expanding the potential industrial applications[5–6]. Among the functional coatings, Y_2O_3 ceramic coating has excellent thermal stability, corrosion resistance, and oxidation resistance[7–8]. Phenolic resin (PF) can improve the hardness, wear resistance, and scratch resistance of the coatings, so it is usually used to cover the surface defects of materials[9]. Therefore, Y_2O_3-PF composite coating can effectively improve the heat resistance, corrosion resistance, and mechanical properties of PMCs, especially for the application in large-scale composite material in aerospace.

In recent years, extensive researches have been conducted on the preparation processes of functional coatings on PMCs, such as chemical vapor deposition (CVD), physical vapor deposition (PVD), impregnation, and thermal spraying[10–13]. CVD method typically requires high temperature conditions and may cause thermal damage and degradation to additive, fiber, and substrate in the composite materials. Due to the mass transfer and reactant diffusion, it is difficult to ensure the uniformity of CVD coating, especially for the large-scale substrate with complex structure[14–15]. For PVD technique, the deposition rate is usually slow, suggesting that more deposition time is required for the preparation of large-scale thick coating. In addition, the coating structure prepared by PVD process is relatively complex, and high-energy ion beam

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bombardment effect forms during sputtering process, which may cause ionized electrons, thus resulting in uneven coating structure and defects[16–17].

Furthermore, the impregnation process requires immersion treatment for substrates, during which the coating thickness is difficult to accurately control. Moreover, the substrate should undergo multiple dipping and drying treatments, which requires long-term preparation and is not suitable for rapid manufacture[18]. In thermal spraying processes, such as flame spraying[19], explosive spraying[20], and plasma spraying[21], high-temperature and high-speed molten droplets tend to cause serious erosion on resin matrix, thereby weakening the bonding strength between coating and matrix. In this research, the supersonic plasma spraying with dual-channel powder feeding technique was used to prepare Y_2O_3-PF alternating coating on PMC surface to obtain enhanced comprehensive performance. Meanwhile, the powder melting deposition behavior under different powder feeding conditions was investigated, and physicochemical tests and analyses were conducted on the as-prepared alternating coatings.

1 Experiment

3240 glass fiber-reinforced epoxy resin composite was selected as matrix material, which was produced by Shenzhen Yongfengyuan Plastic Materials Co., Ltd. Two typical powders were used for spraying, and their morphologies were observed by scanning electron microscope (SEM), as shown in Fig. 1. Y_2O_3-coated PF (Y_2O_3/PF) composite powder with particle size of 0.2 – 115 μm was produced by Beijing Taiyang Spraying New Materials Co., Ltd. The Y_2O_3/PF composite powder has a core-shell structure, which was prepared by agglomeration granulation process, as shown in Fig.2. The spherical Y_2O_3 powder with particle size of 15 – 45 μm was produced by Hunan Jiaoli Thermal Spray Materials Co., Ltd.

The preparation of Y_2O_3-PF alternating coating was conducted by HEP-Jet supersonic plasma spraying equipment, which was developed by National Key Laboratory for Remanufacturing. The supersonic plasma spraying parameters are shown in Table 1. Before spraying, sandblasting pretreatment was conducted on the resin matrix to promote coating deposition and improve interface adhesion. During the spraying process, dual-channel powder feeding technique was adopted, as shown in Fig.3. Y_2O_3 powder was fed radially into high-temperature plasma jet inside the nozzle, whereas the Y_2O_3/PF powder entered the jet from low-temperature zone through external powder feeding mode. To control the temperature of Y_2O_3/PF powder in high-temperature plasma jet and avoid significant erosion of PF particles, an external powder feeder was fixed with the spraying distance of 100 mm from the anode nozzle and 50 mm from the substrate.

Continuous spraying of Y_2O_3/PF powder might lead to the formation of various pores in coating, and continuous spraying of Y_2O_3 powder would burn PF material in resin matrix and coating. Therefore, to avoid the abovementioned adverse situations, Y_2O_3/PF powder was firstly sprayed 5 times on resin matrix as a transition layer. Then, Y_2O_3 powder was sprayed twice on the transition layer. Afterwards, the Y_2O_3-PF alternating coating was prepared by alternating spraying, as shown in Fig.3.

High resolution field emission SEM (Nova Nano SEM50, FEI, USA) was used to analyze the microstructure of powder and coating. Laser particle size analyzer (Mastersizer 3000, Malvern, England) was used to detect particle size distribution. The electron probe X-ray microanalysis (EPMA,

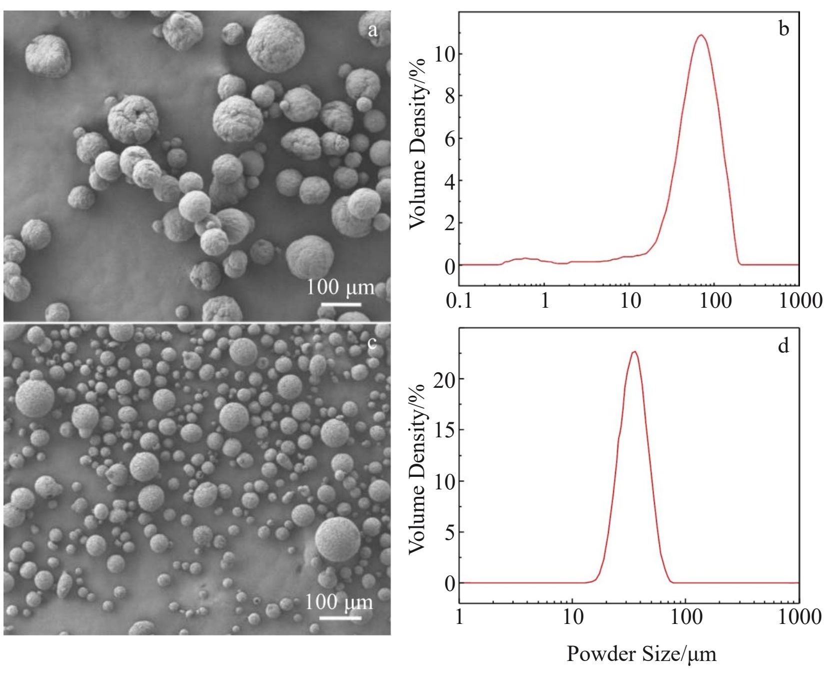

Fig.1 Surface morphologies (a, c) and particle size distributions (b, d) of Y_2O_3/PF composite powder (a–b) and Y_2O_3 spherical powder (c–d)

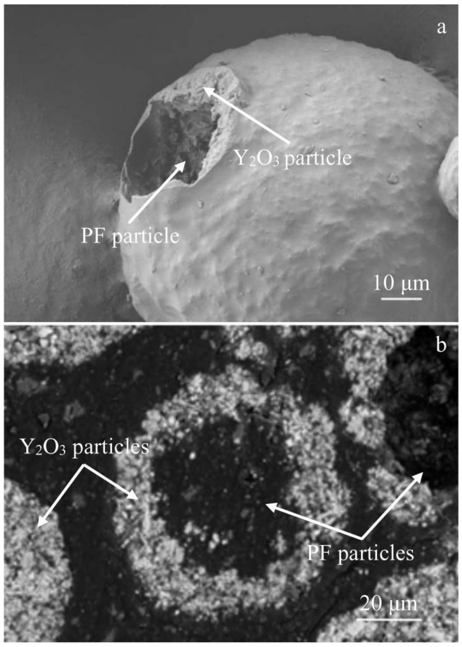

Fig.2 Surface (a) and cross-section (b) morphologies of Y_2O_3/PF composite powder

Table 1 Supersonic plasma spraying parameters

Parameter	Value
Spraying current/A	410
Spraying voltage/V	110
Ar flow/L·min^{-1}	60
H_2 flow/L·min^{-1}	22
Spraying distance/mm	150

JXA-8230, JEOL, Japan) was used to analyze the composition and characteristics of the cross-section of the alternating coating. According to GB/T 8642-2002 and GB/T 33334-2016 standards, the bonding strength and shear strength of alternating coating were measured using a universal testing machine (Exceed E45, MTS, China). When the coating completely or uniformly peeled off, the test data were valid, and the average value of 5 tests was taken as the final result for analysis. In addition, the elastic modulus was measured using a standard micro/nano-indenter (STeP500-NHT3-MCT3, Anton Paar, Australia).

2 Results and Discussion

2.1 Microstructure of Y_2O_3-PF alternating coating

By alternately spraying Y_2O_3/PF and Y_2O_3 powders, the alternating coating is deposited on epoxy resin composite material. The surface morphologies of Y_2O_3-PF alternating coating are shown in Fig.4. During the spraying process, the deposition effect of Y_2O_3-PF powder is prominent, and PF content on coating surface is high, thus resulting in the yellow brown appearance. At the same time, the coating is prepared with obvious particle characteristics, leading to high roughness, as shown in Fig.4a. This is because the spraying is conducted with a small amount of spraying power through long powder delivery process, which can protect the resin matrix and Y_2O_3/PF powder. Therefore, Y_2O_3 with high melting point cannot be fully melted, ultimately exhibiting obvious particle characteristics on the surface. As shown in Fig.4b, unmelted Y_2O_3 particles form small protrusions on the coating after high-speed impact, then accumulate, and grow during the subsequent deposition and stacking process. The well melted Y_2O_3/PF powder is accelerated by jet impact and spreads on the coating surface. The overlapping structure of unmelted Y_2O_3 particles is filled with PF as the binder, eventually forming a relatively smooth protrusion on the coating surface. In addition, in the protrusion area, the Y_2O_3 particles cause a certain degree of ablation during PF filling and bonding, thus presenting the loose structure. On the one hand, according to the size of Y_2O_3 particles in protrusion area, Y_2O_3 powder partially melts in high-temperature jet. During the deposition, the melted part tightly adheres to the unmelted part to form small Y_2O_3 particles (Fig. 4c). On the other hand, in the flat area of coating surface, Y_2O_3/PF powder spreads well without obvious pore structures (Fig.4d).

When the deposition is conducted through alternating spraying, Y_2O_3 particles are evenly distributed on the coating surface and fully bond with PF, thus providing structural support for coating. The cross-section morphologies of Y_2O_3-PF alternating coating are shown in Fig.5, and EPMA results are shown in Fig.6. In addition, obvious pores can be observed in the cross-section of the alternating coating, which mainly exist in the accumulation area of PF material (Fig. 5a). Furthermore, the cross-section morphology of alternating coating clearly shows good adhesion effect with the substrate.

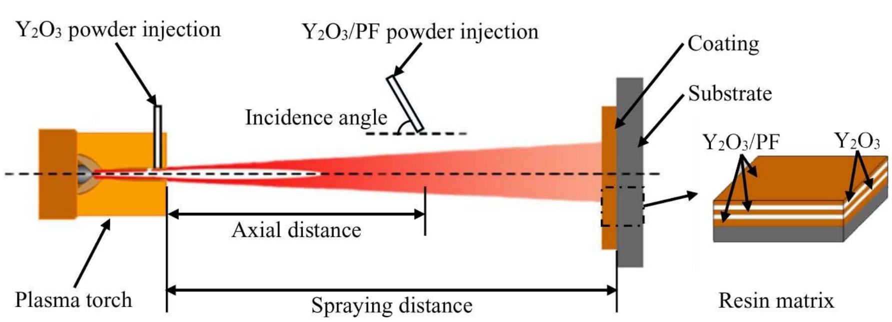

Fig.3 Schematic diagram of dual-channel supersonic plasma spraying process

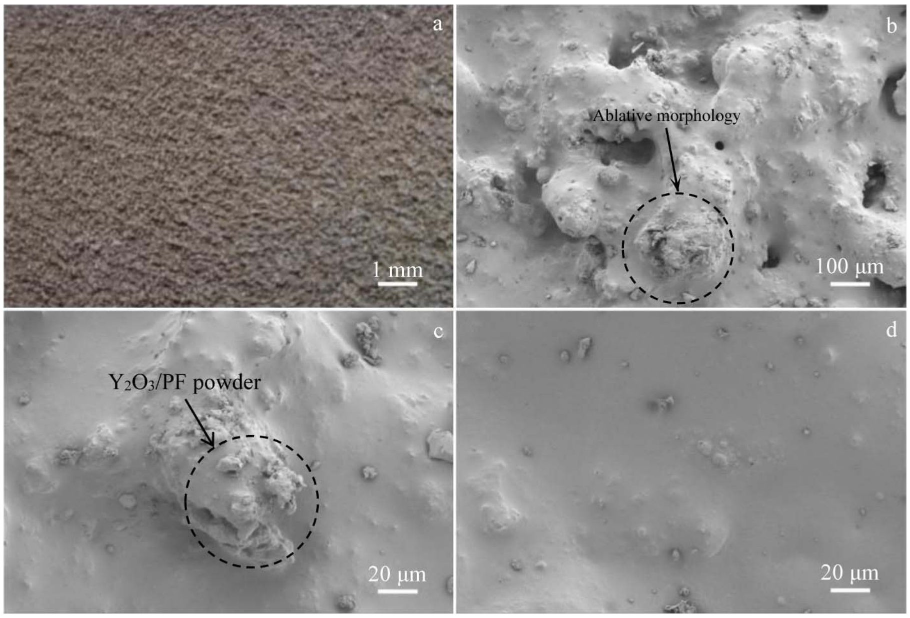

Fig.4 Appearance (a) and SEM morphologies at low magnitude (b) and high magnitude (c–d) of Y_2O_3-PF alternating coating surface

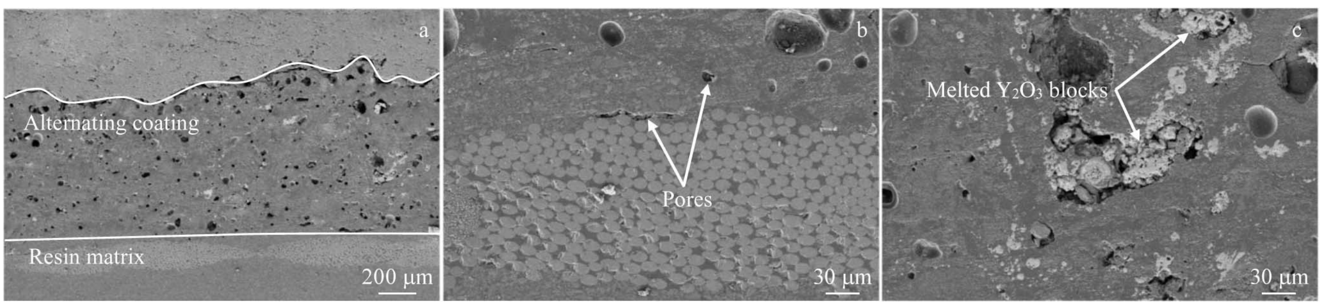

Fig.5 Cross-section morphologies of Y_2O_3-PF alternating coating at low (a) and high (b–c) magnitudes

Although there are a few pores on the bonding interface, no obvious crack defects can be observed (Fig. 5b). Meanwhile, unmelted Y_2O_3 particles gather inside the alternating coating, so PF fully fills the pores between Y_2O_3 particles. These results indicate that the unmelted Y_2O_3 particles accumulate and are stacked during coating deposition, therefore generating abundant pores and cracks and reducing bonding strength between the coating layers (Fig.5c).

2.2 Melting deposition behavior of spraying powder

2.2.1 Melting deposition behavior of Y_2O_3/PF coating with external powder feeding

Fig. 7 illustrates the spreading morphologies of Y_2O_3/PF coating (transition layer) sprayed by external powder feeding. As shown in Fig. 7a, the spreading structure of powder particles exhibits a spreading splashing shape. This is because when Y_2O_3/PF powder enters the plasma jet by external powder feeding, the core PF is protected by the external Y_2O_3 particles and it is heated uniformly during the heating acceleration process, which leads to complete melting. Therefore, after colliding with the substrate at a high speed, PF fully spreads and tightly adheres to the unmelted Y_2O_3 particles. In Fig. 7b, some PF melts and spreads, whereas the majority of powder particles are still aggregated and protrude, thus causing insufficient diffusion of outer Y_2O_3 particles. This phenomenon is caused by the insufficient heat transfer of Y_2O_3/PF powders in the jet. On the one hand, some large-sized particles have higher heat capacity and heat loss, therefore requiring more heat and time for complete melt. On the other hand, in the plasma spraying, the gas around spray gun flows at a high speed and interacts strongly with the powder particles, thus generating a huge shear force which cannot be ignored. As a result, some powders are sheared, rotated, or even refluxed. Therefore, when the heat transfer is insufficient to impact the substrate, Y_2O_3/PF powder will not be able to fully spread and adhere to the substrate, thereby leading to the formation of pores and cracks in the coating.

Fig. 8 shows the microstructures of Y_2O_3/PF coating. It can be seen that the Y_2O_3 and PF exhibit a regional distribution characteristic in the coating, and the fully melted PF adheres to the unmelted Y_2O_3 ceramic particles during the deposition process (Fig. 8a). As shown in Fig. 8b, the ceramic particles and PF on the cross-section form a uniform composite structure, which is uniformly dispersed without obvious interface. The fusion of melted resin and ceramic particles

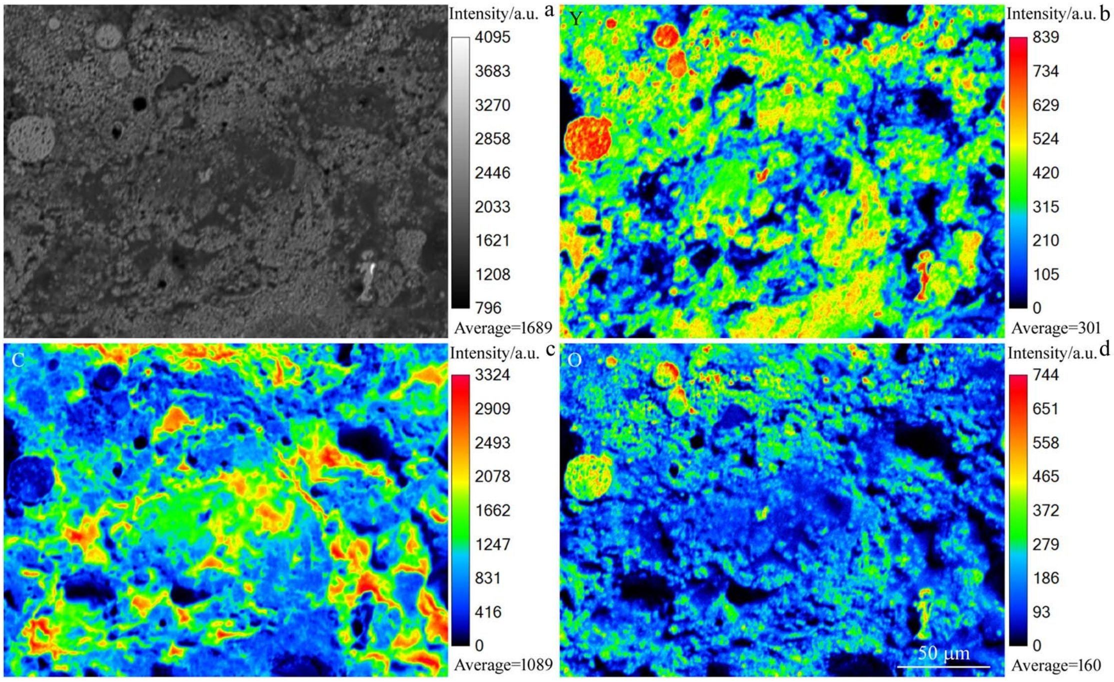

Fig.6 EPMA results of Y_2O_3-PF alternating coating: (a) overall morphology; (b) Y element mapping; (c) C element mapping; (d) O element mapping

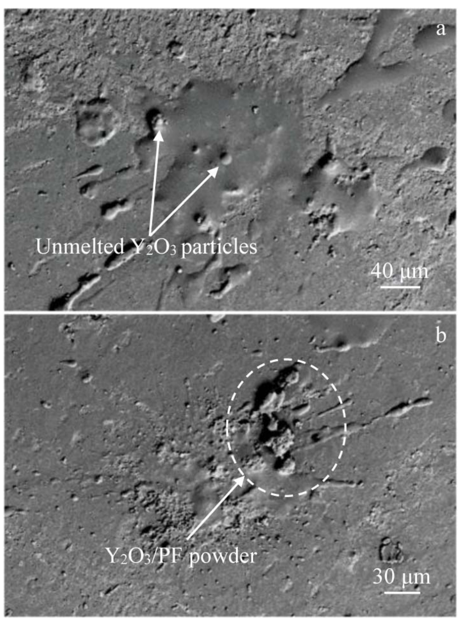

Fig.7 Spreading morphologies of Y_2O_3/PF coating sprayed by external powder feeding at low (a) and high (b) magnitudes

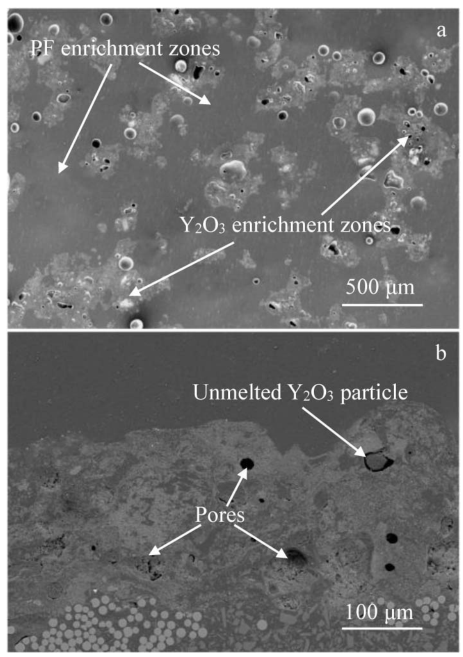

Fig.8 Microstructures of polished surface (a) and cross-section (b) of Y_2O_3/PF coating sprayed by external powder feeding

greatly promotes the compactness and strengthens the coating. The coating usually has good consistency and continuity, and the pores in cross-section are mainly concentrated within PF zones for the following reasons. Firstly, when PF is heated, aldehyde groups react with phenol groups, thus forming cross-linked structures. This process is also called thermal curing reaction of phenolic resin, which will release small molecular gases, including H_2O, as by-products[22]. Secondly, according to the plasma spraying characteristics, it is known that it is impossible to ensure uniform particle accumulation when the particles diffuse and accumulate. At the same time, due to the presence of air pockets inside the particles, a small number of pores are also generated[23]. Thirdly, when the particle melting is not sufficient (Fig. 7b), Y_2O_3 particles may form agglomerations and stacking areas, resulting in uneven particle distribution and size difference, which eventually prevents the complete combination of PF and leads to the formation of pores.

2.2.2 Melting deposition behavior of Y_2O_3 coating sprayed by internal powder feeding

Y_2O_3 powder is also sprayed and deposited onto Y_2O_3/PF transition layer by the internal powder feeding, and the corresponding Y_2O_3 coating morphologies are shown in Fig. 9. It is clear that the impact morphology of Y_2O_3 particles presents an irregular shape, which is similar to the spreading characteristics of liquid. It is also observed that the sprayed Y_2O_3 coating is very rough, and the melted state of Y_2O_3 powder is relatively inferior. Under the action of surface tension and rapid solidification, unmelted Y_2O_3 particles are closely combined with the melted ones, promoting the particle adhesion and close combination with the coating (Fig. 9a). Furthermore, the semi-melted Y_2O_3 particles tend to approach each other and form aggregates, thus providing a deposition platform for the subsequent particles. However, the aggregation of high-temperature particles will lead to resin carbonization, thereby reducing the bonding strength between Y_2O_3 particles and PF. Additionally, the small molecule gas released during the carbonization process will also increase the number of pores in the coating (Fig.9b).

Fig. 10 shows the high-resolution cross-section morphologies of the Y_2O_3 coating, and it can be clearly observed that the coating porosity decreases significantly. This is because the unmelted Y_2O_3 particles produce a shot peening effect during the impact deposition, which enhances the coating compactness and erodes the poorly bonded particles (Fig. 10a). In addition, no obvious cracks can be observed in the bonding area between Y_2O_3 coating and Y_2O_3/PF transition layer. This is due to the deposition of melted Y_2O_3 particles with excessive heat, which leads to the ablation reaction of PF on Y_2O_3/PF transition layer, thus exposing the internal Y_2O_3 particles as a subsequent deposition platform (Fig.10b).

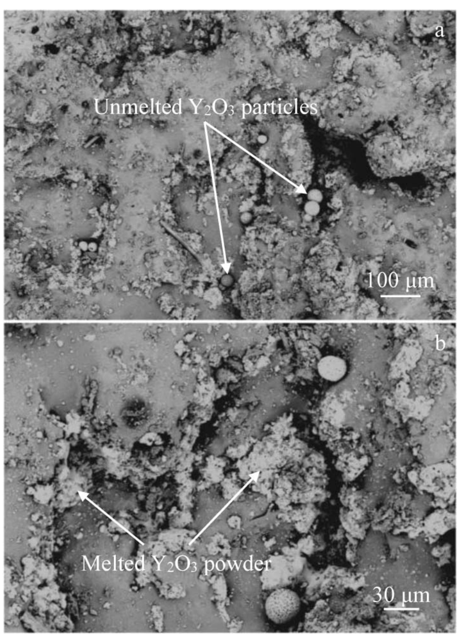

Fig.9 Surface morphologies of Y_2O_3 coating sprayed by internal powder feeding at low (a) and high (b) magnitudes

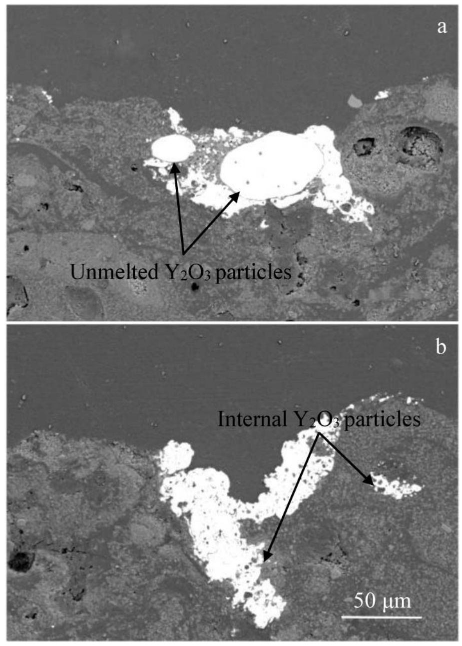

Fig.10 High-resolution cross-section morphologies of Y_2O_3 coating: (a) unmelted Y_2O_3 particles and (b) internal Y_2O_3 particles

2.3 Mechanical properties of Y_2O_3-PF alternating coating

2.3.1 Bonding strength

Bonding strength is an important index to test and evaluate the adhesion states of coatings, including strength, stability, and reliability. The test results of Y_2O_3-PF alternating coating are listed in Table 2: the average bonding strength is 26.48 MPa, and the highest bonding strength of single-test is 28.10 MPa, which is better than the results (below 20 MPa) in Ref. [24]. In addition, the microstructures of Y_2O_3-PF alternating coating after tensile fracture are shown in Fig. 11. It can be seen that the fracture mainly occurs at the interface between alternating coating and resin matrix. At this time, the coating is almost completely separated from the matrix, which indicates that the bonding strength within the alternating coating is greater than that between coating and resin matrix. It is worth noting that some parts of the coating still adhere to the substrate surface, suggesting that the fracture occurs in the alternating coating rather than on the interface between alternating coating and resin matrix. This is due to the inferior bonding strength caused by aggregation and accumulation of some unmelted Y_2O_3 particles during deposition.

Table 2 Bonding strength of Y_2O_3-PF alternating coating (MPa)

Parameter	Value
Bonding strength	27.70, 28.10, 24.40, 26.20, 26.00
Average bonding strength	26.48
Standard deviation	2.09

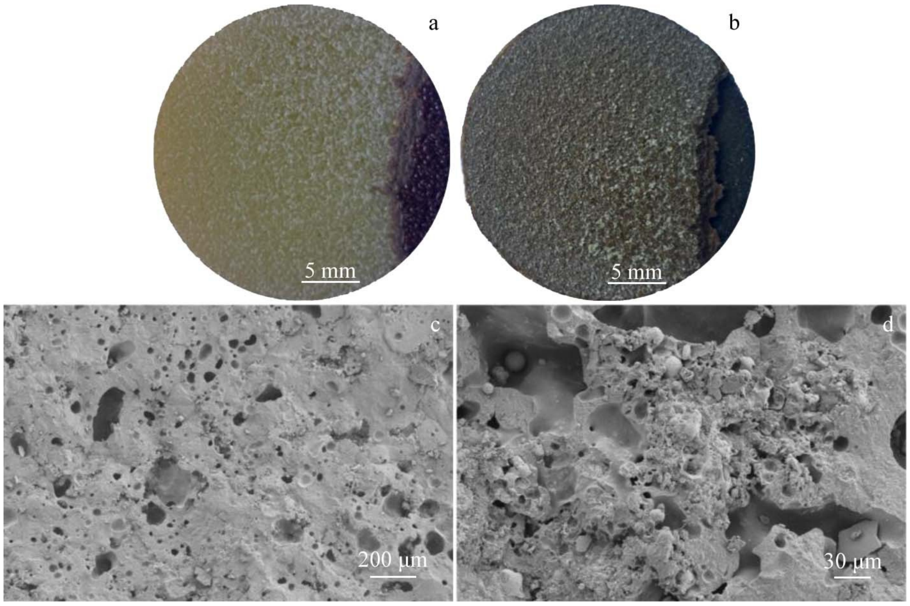

Fig.11 Appearances (a–b) and microstructures (c–d) of cross-sections of matrix (a, c) and Y_2O_3-PF alternating coating (b, d) after tensile fracture

2.3.2 Shear strength

For most parts in the practical applications, shear strength is a necessary mechanical property index for coatings to resist actual mechanical stress. Durability and damage resistance of the coating should also be accurately evaluated by comprehensive consideration of different properties, such as bonding strength and shear strength[25]. Based on GB/T 33334-2016 standard, the shear strength test method of Y_2O_3-PF alternating coating is optimized, as shown in Fig. 12[24]. According to the shear strength test results of Y_2O_3-PF alternating coating in Table 3, the shear strength is basically stable at 24.30 MPa, which is slightly lower than the corresponding bonding strength.

After the shear strength tests, the peeling morphologies of Y_2O_3-PF alternate coating are shown in Fig. 13. Under the action of shear force, the alternating coating almost completely peels off from the substrate surface, which indicates that Y_2O_3-PF alternating coating sprayed by supersonic plasma has a good bonding performance with the resin matrix (Fig. 13a). Based on the microstructure of coating on the aluminum plate (Fig. 13b), PF is broken under shear stress, and the unmelted Y_2O_3 particles almost completely peel off from the matrix but remain in the coating. By analyzing the coating on the aluminum plate in Fig.13c–13d, it can be seen that Y_2O_3 particles cause partial ablation of the bottom resin during the deposition process, which is the fundamental reason for crack initiation under shear stress. In addition, PF as a binder can effectively fill the gap between unmelted Y_2O_3/PF particles, thus improving the shear strength of Y_2O_3-PF alternating coating.

2.3.3 Elastic modulus

Three measuring points were randomly selected for the elastic modulus tests of Y_2O_3-PF alternating coating, and the average elastic modulus is used for analysis. According to the load-depth curves in Fig. 14, the elastic modulus of the Y_2O_3-PF alternating coating is 16.352 GPa, which is close to that of the resin matrix (14.395 GPa). Elastic modulus is an important parameter for stiffness evaluation of materials. When the elastic modulus of Y_2O_3-PF alternating coating is close to that of the resin matrix, the cross-sectional deformation between coating and matrix decreases, which means that the coating bonds more tightly with the substrate. In addition, the coating

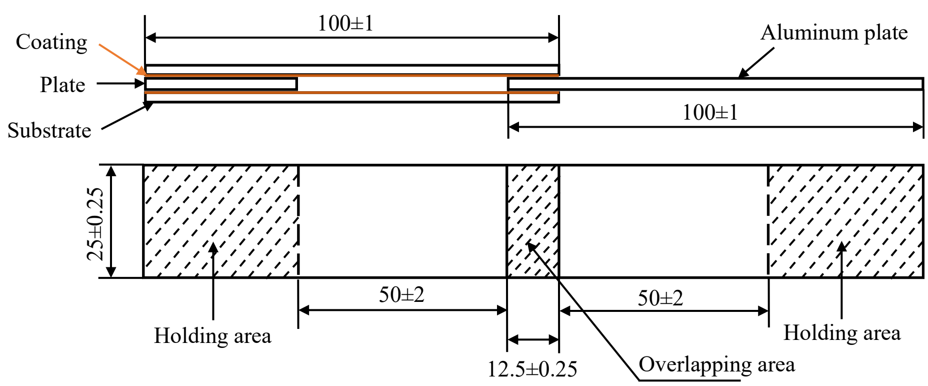

Fig.12 Schematic diagram of double lap shear strength test of Y_2O_3-PF alternating coating[24]

Table 3 Shear strength of Y_2O_3-PF alternating coating (MPa)

Parameter	Value
Shear strength	26.60, 24.50, 24.10, 23.80, 22.20
Average shear strength	24.24
Standard deviation	1.42

can better adapt to the stress environment of the substrate, thus reducing the risk of stress fatigue damage to the coating, which can enhance the durability and anti-damage ability of coating. Therefore, when Y_2O_3-PF alternating coating is under stress, the probability of coating cracking is significantly reduced.

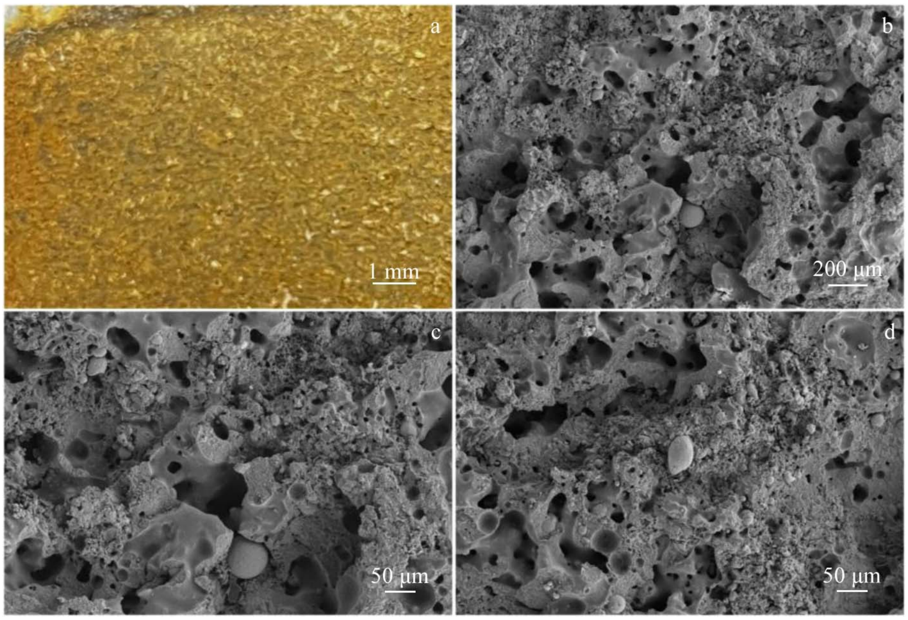

Fig.13 Peeling morphologies of Y_2O_3-PF alternating coating on aluminum plate after shear tests: (a) appearance; (b) SEM image at low magnitude; (c–d) SEM images at high magnitude

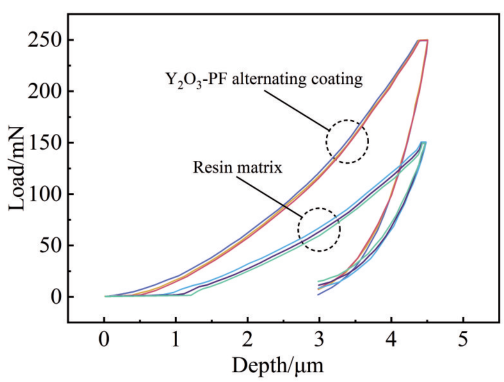

Fig.14 Load-depth curves of Y_2O_3-PF alternating coating and resin matrix

3 Conclusions

1) Y_2O_3-PF alternating coating can be deposited on the surface of 3240 glass fiber-reinforced epoxy resin composite by supersonic plasma spraying. Y_2O_3/PF powder can be sprayed by dual-channel powder feeding method, and Y_2O_3/PF transition layer is formed on the resin matrix. Then, pure Y_2O_3 powder and Y_2O_3/PF powder are alternately sprayed to prepare the Y_2O_3-PF alternating coating.

2) After optimizing the process parameters of supersonic plasma spraying, the average bonding strength of Y_2O_3-PF alternating coating is 26.48 MPa with the single-test maximum bonding strength of 28.10 MPa. In addition, the average shear strength of the coating reaches 24.30 MPa, thus demonstrating excellent mechanical properties.

3) When Y_2O_3/PF powder passes through high-temperature plasma jet, the external Y_2O_3 particles are heated. With the heat transfer, PF is gradually softened and melted, which effectively avoids the damage of high temperature to the molecular structure, thus promoting the cross-linking and solidification of resin matrix during the deposition process. Ultimately, the overall structure and properties of the Y_2O_3-PF alternating coating are improved significantly.

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聚合物基复合材料表面Y_2O_3-PF交替涂层的制备及性能

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摘　要：采用超音速等离子喷涂及双通道送粉技术，在环氧树脂基复合材料表面制备了高性能氧化钇-酚醛（Y_2O_3-PF）交替涂层。将Y_2O_3包覆PF粉末喷涂在基体上生成过渡层，之后交替沉积球形Y_2O_3粉末和Y_2O_3包覆PF粉末形成复合交替涂层。结果表明，交替涂层主体由Y_2O_3包覆PF粉末沉积而成，且涂层与基体结合强度高达26.48 MPa，单次最大结合强度为28.10 MPa，剪切强度高达24.30 MPa。此外，外部Y_2O_3粒子产生的热传递效应使PF逐渐软化熔融，从而有效避免了高温对分子结构的破坏，促进了沉积过程中树脂的交联与固化。同时，未熔Y_2O_3粉末形成了喷丸效应，冲刷去除了沉积效果较差的粉末粒子，最终显著改善了交替涂层的组织和性能。

关键词：Y_2O_3-PF交替涂层；环氧树脂复合材料；超音速等离子喷涂；力学性能

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