Influence of Deposition Temperature on the Splat Bonding and Mechanical Properties of Plasma-Sprayed Tungsten Coatings

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Abstract: The mechanical properties of thermally sprayed metallic coatings are limited by the bonding between splats. In this study, tungsten coatings were deposited at different deposition temperatures by controlling the substrate temperature through shredded plasma spraying. The dependence of the splat bonding and mechanical properties of W coatings on deposition temperature was investigated. The results showed that the apparent porosity of the coatings decreased from 3.2% to 0.3% with the increase of the deposition temperature. The Young's modulus of W coating was significantly increased from 128 to 307 GPa as the deposition temperature increased from room temperature to 800 °C. The microhardness of the coatings was less influenced by the deposition temperature. It was found that splat bonding across lamellae was formed when the deposition temperature was higher than 600 °C compared to the obvious lamellar interface in the coatings deposited at temperatures lower than 600 °C. The results evidently revealed that the mechanical properties of plasma-sprayed W coatings could be controlled through the splat bonding by altering deposition temperature.

Key words: plasma spraying; tungsten coatings; deposition temperature; splats bonding; hardness; elastic modulus

Tungsten (W) is a most important industrial material, which is widely used in the fields of wear resistance, are generating and plasma-facing armor material, due to its advantages such as high hardness, high melting point, low tritium inventory and low erosion rate under plasma loading[1-4]. However, the high ductile to brittle transition temperature (approximately 400 °C) and difficulties in machining make it more suitable to be used in form of a coating on a substrate of another ductile material. Up to now, various approaches, such as chemical vapor deposition[5], magnetron sputtering[6,7], arc-deposition[8] and plasma spraying[9], were widely utilized for the deposition of W coatings. Among those deposition methods, plasma spraying is characterized as a fast, low cost and flexible controllable technology, which attracts intensive research and industrial attentions in the past two decades.

Plasma-sprayed coatings are composed of splats or lamellae created by the rapid solidification of molten metal and/or semi-molten feedstock particles on a surface which can be either a substrate surface or the surface of the previously deposited coatings. The coatings is composed of many flattened splats, 1 μm to several micrometers thick and 50 μm to several hundred in diameter, which are bonded together by a limited interface area between lamellae[8]. Due to high amount of the pores in the coating, many properties, such as elastic modulus, fracture toughness, thermal and electric conductivity, of plasma-sprayed coatings are found to be as low as 1/10-1/3 of those of bulk material[10-12]. Although it is ever attributed to the high porosity of the coatings, the much lower properties can not be well explained by the conventional theories for typical porous materials.

It is worthy to note that the pores in plasma-sprayed coatings are not homogeneously distributed in the coatings but of a significantly heterogeneous characteristic[10,12,13]. The pores of plasma-sprayed coatings exhibits a bimodal distribution, with coarse pores in the size range of 3-10 μm and fine ellipsoid pores having a width (minor axis) of about 0.1 μm[12,13]. The coarse pores with a near-spherical morphology result from incomplete filling of interstices between previously deposited particles by impacting melted particles. The fine pore is found in the form of narrow non-contact gaps between the splats mainly in the direction parallel to splash surface, although that in ceramic coatings also includes vertical microcracks within the splats (intralamellar cracks) which are generated by quenching, a lack of plasticity of the ceramic mate-

Received date: May 25, 2011
Foundation item: National Natural Science Foundations of China (50725101)
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rial, and subsequent stress relaxation during the cool-down process.\textsuperscript{[14]} McPherson proposed a model for the relationship between the microstructure, in which non-bonded area is emphasized, and the physical and mechanical properties of plasma-sprayed coatings\textsuperscript{[9,10]}. Since the successful quantitative characterization of the porous microstructure characteristics including the lamellae bonding ratio was reported by our previous results\textsuperscript{[13]}, it becomes more and more clear that the limited bonding ratio between lamellae is the dominant factor for the much lower property of the coatings compared to those of bulk material.

For plasma-sprayed W coatings, vacuum plasma spraying or low pressure plasma spraying are proposed to deposit high performance W coatings by lowering the oxidation of W splats\textsuperscript{[15-18]}. Although many investigations show that the splat bonding is influenced by many factors such as the temperature and velocity of spray particles, substrate temperature and vacuum chamber pressure, the bonding between the splats reported up to now was still much limited\textsuperscript{[19-23]}. Accordingly, it was also observed that those physical and mechanical properties of plasma-sprayed W coatings were much lower than those of bulk W material\textsuperscript{[21-27]}. Our previous results proposed that a high bonding ratio could be evidently realized by increasing the deposition temperature to a high level of 400-800 °C for Al₂O₃ coatings and 600-1100°C for YSZ coatings\textsuperscript{[28-30]}, although the detailed mechanism is still far away from fully understanding.

In this study, the dependences of the microstructure and properties on the deposition temperature of plasma sprayed metallic W coatings were investigated, aiming at contributing to the comprehensive understanding of the relationship between the deposition temperature and the splat bonding.

1. Experiment

Commercially available W powder with a size range from 32 to 84 μm was used for the deposition of W coatings on copper substrates (Fig.1). The copper substrate was employed for precisely controlling and monitoring substrate surface temperature during coating deposition. A commercial plasma spraying system (80 kW) was employed to generate an Ar-H₂ plasma jet at a plasma arc power of 36 kW to melt spray powder particles. To lower the oxidation of the W particles, a nitrogen gas shrouding attachment in a diameter of 50 mm and length of 122 mm was installed on the plasma torch. The flow rate of the protective N₂ gas was fixed at 40 L·min⁻¹. The spray distance from the nozzle exit to the substrate surface was 125 mm. The plasma torch was manipulated by a robot to traverse spray particle streams over the substrate perpendicularly. The surface temperature of copper substrate was monitored by k-type thermocouples.

The cross-sectional microstructure and surface morphology of W coatings were examined by scanning electron microscopy. Vickers micro-hardness of the coating was measured on a polished cross-section at a load of 2.94 N and a loading time of 15 s. The Young’s modulus was measured through the Knoop indentation approach as proposed in the literature\textsuperscript{[31]} at a load of 2.94 N and a loading time of 15 s.

2. Results and Discussion

2.1 Effect of deposition temperature on coating microstructure

Fig.2 shows the cross sectional microstructures of plasma-sprayed W coatings deposited at different deposition temperatures from 60 °C to 800 °C. Some near-spherical pores in a size of several micrometers were observed in the coatings. In addition, a few relatively large non-bonded lamellar interface areas were present in the coating deposited at a deposition temperature of 60 °C. Since the narrow non-bonded areas can not be clearly recognized at the magnification of the image in Fig.2, most of the apparent pores are possibly induced during the polishing process of the sample. To quantitatively compare the pores of the coatings, the apparent porosity of the coatings was estimated based on image analyzing using at least 5 images for each deposition temperature. As shown in Fig.3, the apparent porosity of the coatings significantly decreased from 3.2% to 0.3% with the increase of the deposition temperature from 60 °C to 800 °C. This fact suggests that the bonding between lamellae becomes stronger with the increase in the deposition temperature, which reduces significantly apparent pores resulting from pulling out during sample preparation.

2.2 Effect of deposition temperature on mechanical properties

Fig.4 shows the effect of deposition temperature on the micro-hardness and elastic modulus of plasma-sprayed W coatings. The microhardness of the coatings slightly increased from 4000 to 4700 MPa with the increase of deposition temperature. It can be clearly found from Fig.4b that the elastic modulus of the coatings was significantly increased with the increase of deposition temperature. When the deposition temperature was maintained to be 60 °C, the elastic modulus of 128 GPa obtained was consistent with the data reported for atmospheric or vacuum plasma sprayed coatings in the published literature\textsuperscript{[24-26]}. However, the

![Morphology of the tungsten powder used for coating deposition](image-url)
much higher elastic modulus of 307 GPa of the coating deposited at a deposition temperature of 800°C became three-fourth of the bulk W material, i.e. 410 GPa. Such significant increase in Young’s modulus of plasma-sprayed W coating is attributed to the significant increase of the lamellar interface bonding in the coating deposited at a higher deposition temperature.

2.3 Effect of deposition temperature on splats bonding

To reveal the bonding condition between the W splats in the coatings, the cross-sectional morphologies of fractured coatings were examined and shown in Fig.5. All the coatings exhibited a typical lamellar structure in which the columnar grains perpendicular to lamellar interface having a diameter of 0.5-1 μm were clearly observed in individual lamellae. The thickness of 4-8 μm of individual splats was evidently not significantly influenced by the deposition temperature. It can be found that the non-bonded areas between the splats can be clearly recognized in the coatings deposited at relatively low deposition temperatures (Fig.5a-5b). However, when the deposition temperature was increased to 610 °C (Fig.5c-5d), the bonding between lamellae became much improved and few non-bonded areas could be recognized. In addition, it can be clearly recognized that the columnar grains in individual splats grew across adjacent splats in the direction perpendicular to the lamellar plane, which makes individual splats be well bonded and thus less distinguishable.

The elastic modulus of plasma-sprayed coatings is widely reported to be as low as 1/10-1/3 of bulk materials owing to the limited bonding ratio, which is estimated to be less than 32% for plasma-sprayed ceramic coatings. The lower modulus of the plasma-sprayed W coatings is also attributed to the oxidation of W particles, which is supported by the fact that W oxides...
Fig. 5: Morphologies of cross-sections of fractured tungsten coatings deposited at different deposition temperatures: (a) 60 °C, (b) 240 °C, and (c, d) 610 °C

are experimentally found between W splats. Vacuum plasma spraying is highly proposed to deposit high performance W coatings with low oxygen content of less than 0.1%. However, even for the vacuum-plasma-sprayed coatings, the reported elastic modulus of 54-126 GPa is much lower than that of bulk W. Boulos and coworkers reported the increase of the compressive modulus of W coatings to 230 GPa with the increase in the substrate temperature from 300 to 564 °C by using the optimized processing parameters through induction plasma deposition technology. The higher modulus was attributed to the increase of the splat bonding resulting from the change of the splashing splats to the regular disk-shape morphology. The splat morphology transition from splashing to disk-shape owing to adsorbate desorption with the increase in the substrate temperature was also confirmed by our previous study and other literature. However, a typical lamellar microstructure can be distinguished from the images in literature.

Compared to those reported literature, the much higher elastic modulus of 307 GPa, which is 3/4 of the bulk W, clearly shows the significant influence of the deposition temperature on the splat bonding condition. According to the previous studies, the Young's modulus of plasma-sprayed coating is proportional to the lamellar interface bond ratio and the modulus of bulk material when the bonding ratio is higher than about 40%, since the bending effect of lamella during transfer of load can be neglected. The lamellar interface bond ratio in the coatings deposited at 610-800 °C is estimated to be 71% and 75%, respectively. This estimation result is well consistent with the enhanced bonding observed from cross-section of fractured coating as shown in Fig. 3c-d, exhibiting many well-bonded individual splats grown across splat interfaces in a direction perpendicular to the lamellar plane. Taking the significant bonding enhancement of plasma-sprayed YSZ and Al₂O₃ ceramic coatings at elevated deposition temperature into consideration, based on the present results of the improvement of the splat bonding and elastic modulus of the plasma-sprayed metallic W coatings, it can be evidently concluded that the lamella bonding of metallic coatings can be controlled to a relatively high level by the deposition temperature in the same way as that proposed for ceramic coatings. Since most of the properties of the plasma sprayed coatings can be effectively controlled by the lamella bonding condition, the further deeper understanding of the formation mechanism of the bonding at elevated deposition temperature would be expected to aim at a more flexible control of the microstructure and properties of plasma sprayed coatings.

3 Conclusions

The dependence of splat bonding and mechanical properties of plasma-sprayed W coatings on deposition temperature was investigated. The apparent porosity of the coatings decreased from 3.2% to 0.3% with the increase of deposition temperature, indicating the improvement of the splat bonding. The elastic modulus of W coating was significantly increased from 128 to 307 GPa as the deposition temperature increased from room temperature to 800 °C. The microhardness of the coatings was 4000-4700 MPa and was less influenced by the deposition temperature. It was found that the columnar grains in individual splats grew across the interface of multiples of splats in the direction perpendicular to the lamellar plane when the deposition temperature was reached to higher than 610 °C, which made the lamellar interfaces less distinguishable comparing to the obvious lamellae interface in the coatings deposited at lower temperatures. The lamella bonding ratio was estimated to be as high as 75% for the coatings deposited at a deposition temperature of 800 °C based on the reported model. Evidently, the mechanical properties of plasma-sprayed W coatings can be effectively controlled through the splat bonding by altering deposition temperature.

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