Microstructural Characteristics and Wear Performance of Plasma Sprayed Al_2O_3-13 wt. % TiO_2 Coating on the Surface of Extrusion Wheel

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Abstract: The conventional Al_2O_3-13 wt. % TiO_2 composite ceramic coatings are fabricated by plasma spraying on the surface of extrusion wheel. The microstructure, morphology and phase compositions of the substrate and coating are investigated by using X-ray diffractometry (XRD), scanning electron microsopy (SEM) and energy dispersive spectroscopy (EDS). Moreover, the microhardness of the substrate and the coating are investigated using Vickers microhardness tester, the friction and wear behaviors of the substrate and the coating are investigated by using a block-on-ring tribometer under dry sliding conditions with the load of 245 N. The results show that both γ-Al_2O_3 and α-Al_2O_3 phases are observed in the as-sprayed coatings, the mian phase is γ-Al_2O_3. There are white particulates Al_2O_3 on its surface. The Al_2O_3-13 wt. % TiO_2 coating possesses higher microhardness which is about 1018HV and 1.6 times that of the substrate. The wear performance of coating is better than that of the substrate. In a practical application, the life of the extrusion wheel which is plasma sprayed Al_2O_3-13 wt. % TiO_2 coating on the surface is 1.2 times that of the conventional extrusion wheel, and the life is about 330 h.

Key words: extrusion wheel; plasma spraying; Al_2O_3-13 wt. % TiO_2 ceramic coating; wear performance

1 Introduction

Extrusion wheel is one of the main parts of continuous extrusion machine. In the working process, extrusion wheel will bear combined effects, such as friction torque, thermal stress, high temperature and high pressure, and soon [1,2]. The desined design life of the extrusion wheel is 200 h, however the extrusion wheel lose efficacy, which is always used only about 150 h. There are two main problems, one of them is fracture failure, transverse cracking mainly along the bottom of extrusion wheel and radial fracture mainly along the root of spline tooth in the wheel center. By the reason of them is the cracking on the surface of the wheel expanding rapidly [3,4], the other is wear-out failure, which is due to the effect of mechanical stress and thermal stress, then on the groove surface of extrusion wheel, there is producing a flake. All of them are affecting production efficiency and input cost of the enterprises.

Theoretically, plasma-sprayed Al_2O_3-13 wt. % TiO_2 coatings on the surface of extrusion wheel can improve its characteristics, and it is a kind of effective method to improve the servicing life of the extrusion wheel. Plasma spraying is one of the most widely used in the thermal spraying, which possess high flame temperature, fast deposition rate, high efficiency and wide application, and soon [5,6]. Al_2O_3 coatings with TiO_2 has high strength and hardness and good toughness, and is applied to reinforce and protect the working parts, can greatly improve its servicing life. Since the 1950s, the application in the field of aircraft engine has extended to machinery and conveying with good abrasion resistance, as well as the chemical and metallurgical industries with strong erosion resistance, and soon [7,8]. The microstructure compositions and the defects of surface morphology on the surface of Al_2O_3-

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13 wt. % TiO_2 coatings, will have important influences on the hardness and wear resistance, and also influence the servicing life of the extrusion wheel directly, which need further study. Therefore, in this paper, the surface morphology of AT13 coatings which before and after test are observed, the microstructure of coatings is analysed, as well as the wear resistance is studied, which are provided the modification of the surface with experimental proofs.

2 Experimental procedure

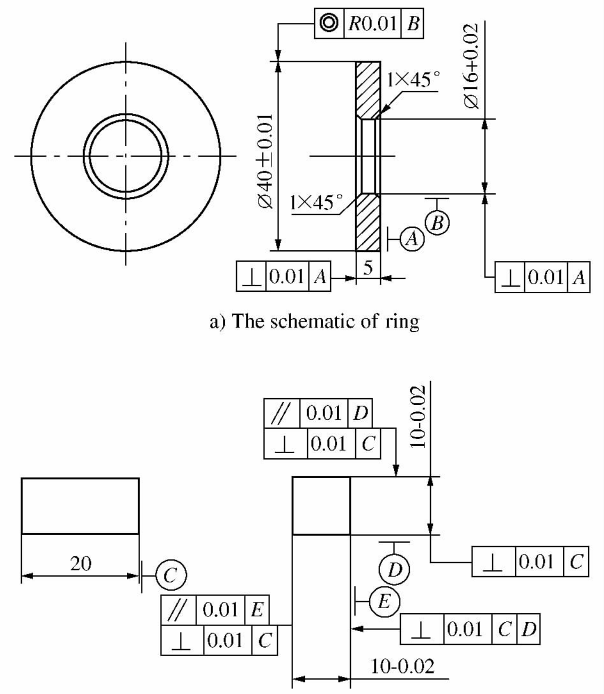
In this work, a commercial alumina powers of 13wt. % TiO_2 (hereafter referred as AT13) with a mean diameter of 30 μm were used as raw material. The thickness is about 300 μm to 400 μm. To reduce the thermal-physical property difference between AT13 coating and substrate material, a bond coating, made of Ni/Al powders, with the thickness between 100 μm and 150 μm. H13 is chosen as the substrate material, which is cut into coupons in a size of 10 mm × 10 mm × 10 mm by means of wire electrical discharge machining. Firstly, to grain refinement of H13 adequately, forged H13, then preheated with the different temperature of 680 ℃ and 820 ℃, after that H13 should be through-hardened by oil quenching at a temperature of 1 050 ℃ and tempered twice (2 hours each time) at a temperature of 620 ℃, the Rockwell hardness of H13 is in the range of 42 to 48HRC. At last, H13 should be pre-processed before plasma spraying. In addition to remove oil of the surface, using acetone, then drying it, coarsening it with sand blasting. Plasma spraying is performed using a Praxair atmospheric institution with manual console type CNC4500. The atmospheric plasma spraying parameters are listed in Table 1.

Table 1 Atmospheric plasma spraying parameter

Spraying parameters	Bond coating	Ceramic coating
Current	680 A	720 A
Voltage	70 V	37.5 V
Primary gas, Ar	96 Scfh	92 Scfh
Secondary gas, H_2	13 Scfh	15 Scfh
Powder carrier gas, Ar	9 Scfh	9 Scfh
Powder feed amount	17 g/min	20 g/min
Spray distance	110 mm	100 mm

The phase composition of the feedstock and the coating are studied by using X-ray Diffractometry (XRD, D/Max2500PC). The surface, fracture surface and the chemical element distribution of the coating are inspected with Scanning Electron Microscopy (SEM, JSM-6360LA) and Energy Dispersive Spectro-scopy (EDS). The microhardness measurements are conducted on the surface of the coating using the HV-1000 Vickers Microindentor with the loads 50 N applied respectively for 20 s. Then taking the average of the 5points as the result. The tribological properties are measured using a M200 Metal abrasion tester under unlubricated sliding contact against the steel ring (45 steel, in Figure 1a) and with the loads of 245 N

(25 kgf) applied respectively for 2 h. The tests are conducted at room temperature and lab air environment. The block (H13 or AT13 coating, in Figure 1b) at a standstill, the ring rotates at a speed of 185 r/min which is shown in Figure 2. The sliding velocity of the block is 0.387 m/s. Prior to the test, the surface of the coating is polished. For wear mechanisms determination, reading the stable friction coefficient, weighing the weightlessness after 2×10^4 r, examining the worn surfaces with the mean of SEM.



a) The schematic of ring

b) The schematic of block

Figure 1 The schematic of block and ring test specimens

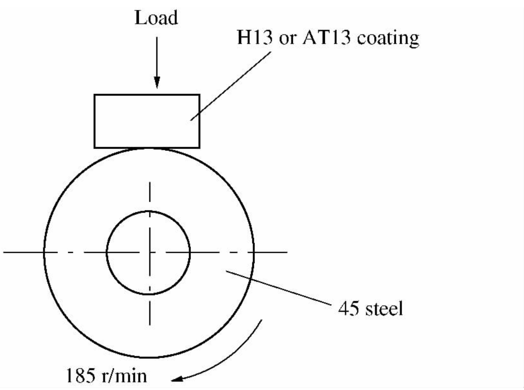
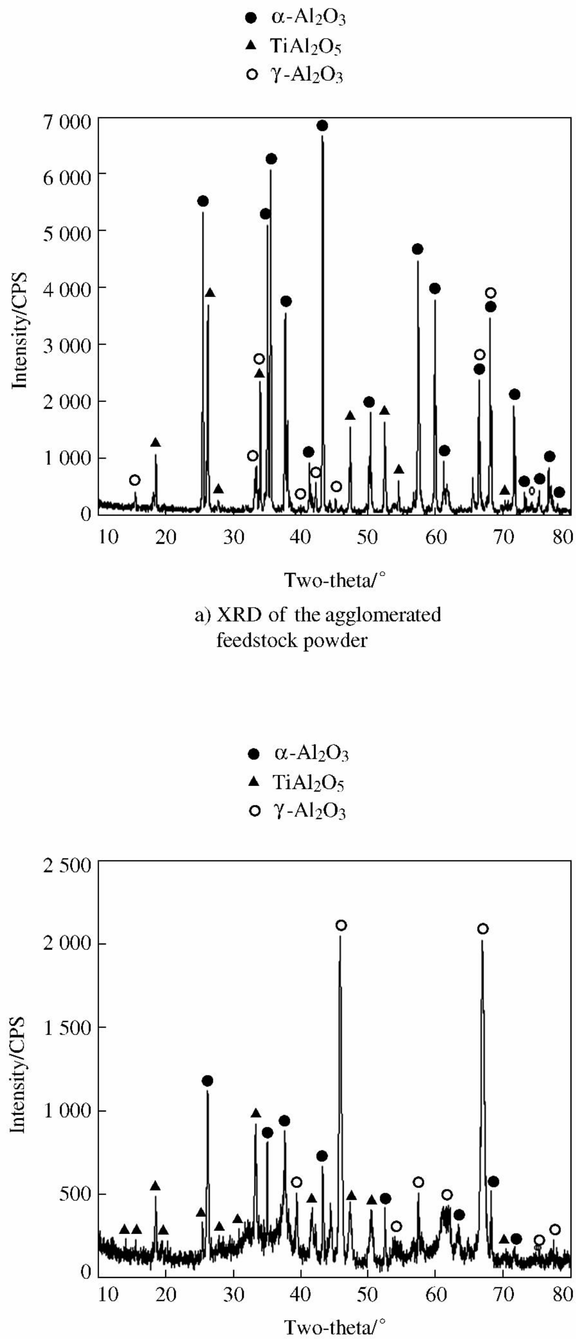


Figure 2 The schematic of block-on-ring arrangement in wear test

3 Results and discussion

3.1 Phase composition of the agglomerated feedstock powder and as-sprayed coating

Figure 3 shows XRD patterns of the agglomerated feedstock powder and AT13 coating, which reveal a similar phase composition for them.



a) XRD of the agglomerated feedstock powder

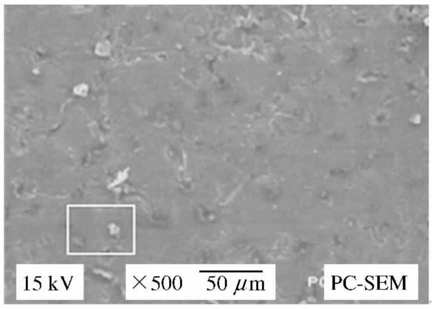
b) XRD of the AT13 coating

Figure 3 XRD of the agglomerated feedstock powder and AT13 coating

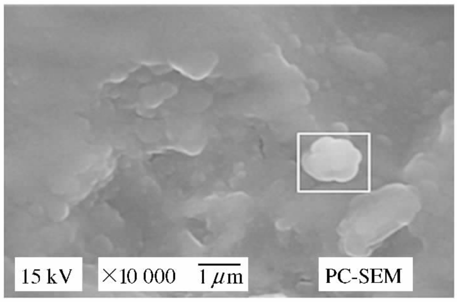
There are two forms of ingredients in the sintered agglomerated powders as show in Figure 3a), α-Al_2O_3 and two modifications of spinel $TiAl_2O_5$ are the main phases. In addition, there is a small quantity of γ-Al_2O_3 that exist in the sintered agglomerated powders. It is obviously seen that AT13 powder is sintered in the proportion of 87:13(Al_2O_3:TiO_2). Al_2O_3 and TiO_2 are easily converted into a stable solid solution at high temperature. In addition, there is a small quantity of γ-Al_2O_3. There are three forms of ingredients in the as-sprayed AT13 coating as show in Figure 3b), γ-Al_2O_3、$TiAl_2O_5$ and α-Al_2O_3, among them γ-Al_2O_3 is the main phases, the two strongest peaks in the picture were γ-Al_2O_3 and the second strong peaks are α-Al_2O_3. The XRD analysis shows that some of α-Al_2O_3 in the powder changes to γ-Al_2O_3 after plasma spraying process. It is well established that because of a higher cooling rate γ-Al_2O_3 commonly nucleates in preference to α-Al_2O_3 [9]. In addition, some of α-Al_2O_3 in the powder are not melt or partly melt, then they still exist in the as-sprayed coatings.

3.2 Surface morphology of the as-sprayed coating

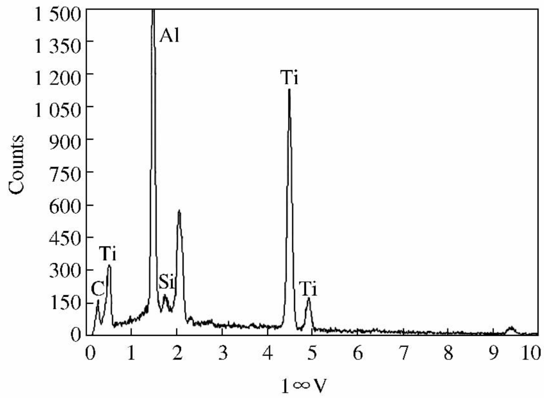
Figure 4a) presents the surface of the plasma-sprayed AT13 coating. The surface of coating is grey. Most of the powders are formed into a dense, flat coating under high temperature flame flow. There are some white particulates on the surface as show in Figure 4b), with the chemical element mass fraction are: 7.83% C, 29.11% O, 61.97% Al, 0.35% Si, 0.74% Ti; and the molar ratio are 13.59% C, 37.94% O, 47.89% Al, 0.26% Si, 0.32% Ti, which is shown in Figure 4c). Form these date above, Al and O elements possess the greatest proportion, then we can conclude that the white particulates are Al_2O_3 which do not melt completely, that's because the melting point of Al_2O_3 is much higher than the temperature of plasma spraying. O element content is lower, the reason is that oxide loss of oxygen under high temperature. Thus the C and Si elements are form the surface of H13, which are melted on the surface of coating under high temperature.



a) SEM micrographs of the surface of AT13 coating



b) Magnification of Figure 4a)



c) EDS analysis of the white granular

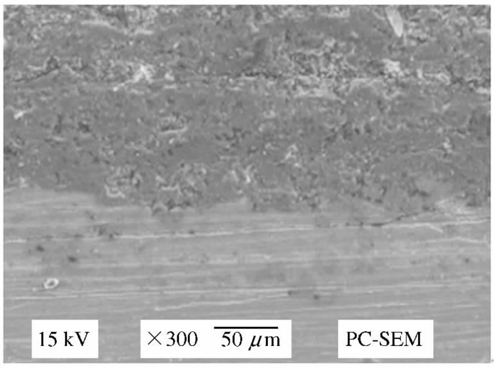
Figure 4 Micrographs of AT13 coating and EDS analysis of the white granular

3.3 Cross-sectional morphology of the as-sprayed coating

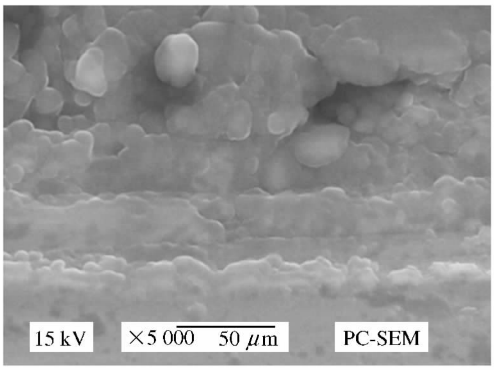
Figure 5a) presents the cross-section of the plasma-sprayed AT13 coating. It can be seen that the as-sprayed ceramic coating has a typical lamellar stacking characteristic. Form up to down, there are the AT13 ceramic coating, bond coating and H13 substrate in turn. The ceramic coating consists of both fully melted regions and partially melted regions, which combines with bond coating closely, as well as the bond coating and H13 substrate. In the ceramic coating existing some small black pores. The reasons of these pores as follows:

1) Al_2O_3 has a high melting point which is about 2 050 ℃, can not be fully melted in the plasma flame flow [16].

2) The other is that air around the substrate will be closed in the ceramic coating as soon as the coating crystallizes which has no time to escape in the process of plasma, with the rise of temperature air escaping from the coating, then there will be small pores left. Detailed SEM examination at high resolution of the ceramic coating is shown in Figure 5b). The coating presents an obvious layered structure and each layer bounds tightly.



a) Overall morphology



b) High magnification of AT13 coating

Figure 5 Cross-sectional morphology of AT13 coating

3.4 Microhardness of the substrate and AT13 coating

The microhardness of substrate and AT13 coating are shown in Figure 6. It can be seen that the AT13 coating possesses improved hardness than substrate. The microhardness of substrate is in the range of 610HV to 625HV, and the average microhardness is about 618HV, while the coating is in the range of 1000HV to 10050HV, the aveage is aout 1018HV. It is obviously seen that the microhardness of coating is about 1.6 times than the substrate, it is because the intrinsic of material determines the hardness, and there are a lot of oxides in the coating which can improve the hardness of coating, in other words, improve the hardness of extrusion wheel.

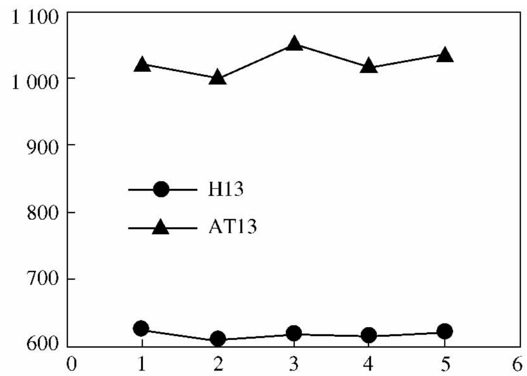


Figure 6 Microhardness of the substrate and AT13 coating

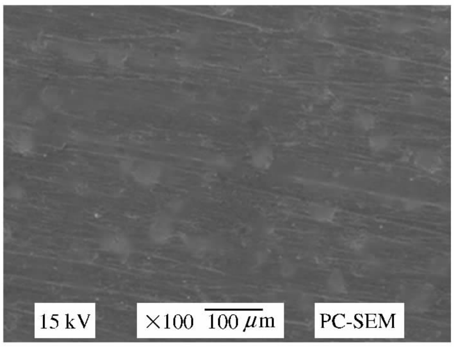
3.5 Friction and wear tests

Prior to the test, the substrate and coating are ground using grit SiC papers and then polished using diamond slurries down to a mean surface roughness of 0.1 to 0.4 μm. During the test, the friction coefficient undulates drastically at the beginning, then it will stable gradually after 2 hours. The tests are conducted at room temperature and lab air environment. As listed in Table 2, the friction coefficient of substrate is lower than the coating, the reason is that the coating consists of both unmelted Al_2O_3 particulates and partially melted Al_2O_3 particulates, which can influence the friction coefficient of coating during the test. The principles of extrusion wheel is that the blank can be squeezed in the extrusion wheel with the friction between groove and blank. The coating on the surface of extrusion wheel possesses improved wear performance than the substrate, and the abrasion loss of the coating is lower than the substrate. In this way, the extrusion wheel's life will be improved.

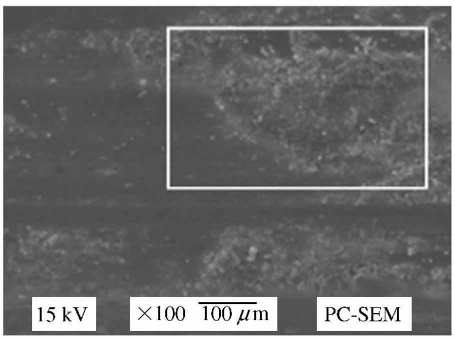
Table 2 The friction coefficient and abrasion loss of the substrate and coating

Sample	Abrasion loss/g	Friction coefficient μ
AT13	0.005	0.48
H13	0.018	0.32

Figure 7a) and Figure 7b) respectively show the microstructure of the surface and worn surface of H13 substrate. For the surface of substrate, there exists some obvious fibrous scratch. From the Figure 7b), it can be seen that there are obvious mild adhesive wear and spalling on the worn surface of substrate, with the roation of the ring, the Al_2O_3 particulates which is prominent to the surface of substrate rub against the ring, and the contact point will occurre adhesive wear when the local stress of contact point over a certain point. The material on the surface which is torn and peeled off will bring great impact on the structural stress and also reduce the wear resistance of the substrate during the wear test.

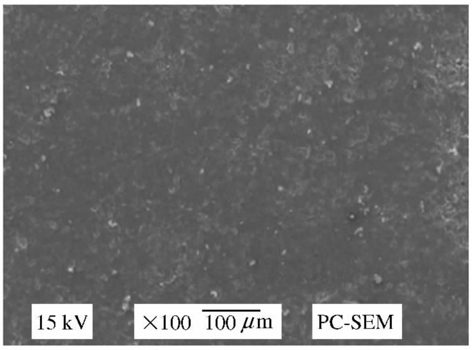


a) SEM micrograph of the surface of H13 substrate

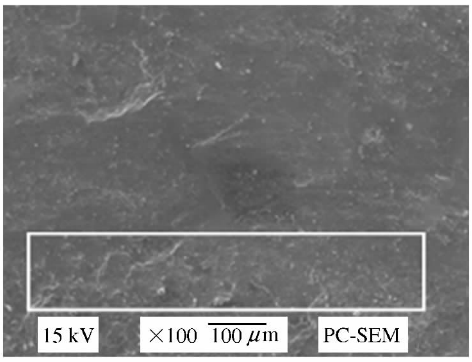


b) SEM micrograph of the worn surface of H13 substrate

Figure 7 Morphology of the H13 substrate before and after wear



a) SEM micrograph of the surface of AT13 coating



b) SEM micrograph of the worn surface of AT13 coating

Figure 8 Morphology of the AT13 coating before and after wear

Figure 8a) and Figure 8b) respectively show the morphologies of AT13 coating and worn surface of AT13 coating. Some white Al_2O_3 particulates on the surface of AT13 coating which is not completely melt is shown in Figure 8a), the smooth surface has no obvious scratches and raised parts. From the Figure 8b), there are obvious bedseparation without an accumlation of wear debris. Due to the consist of both semi-vitreous Al_2O_3 particulates and partially melted Al_2O_3 particulates in the coating, these particulates can reduce the plough effect of 45 steel on the coating which can play the function of toughening, however they can make the surface of coating irregularity and mile adhesive wear. Plastic deformation can occur in the ledge of coating and the areas of the ring with abrasion under the pressure of 245 N. Fracture offen occurs in the ledge of coating where the stress is relatively weak, then some semi-vitreous Al_2O_3 will flake away from the coating. Under the pressure of 25 N, abrasive dusts are not hard enough to resist the crush and broken into tiny particles which can share the load evenly. As a result, chunks of wear debris can not be left on the surface of coatig. On the surface of coating, there is no obvious cracks, it can be seen that the coating has high toughness, which can improve the life of extrusion wheel.

The breaing-off of coating can be seen from Figure 8b). The reasons of it as follows:

1) The surface of coating will produce adhesive wear during the wear process, and the coating can be stear off or tear up in the relative tangential movement between the ring and the coating, then peeled off gradually.

2) In the coating, there are semi-vitreous Al_2O_3 particulates and partially melted Al_2O_3 particulates, which can loose and pull off with the cutting and rotation of the ring during the wear process. Some small cracks will be formed on the surface of coating, with the crack propagation, the coating will peel off.

3) The as-sprayed conventional ceramic coating has a typical lamellar stacking characteristic, some small pores will exist between the layers which is very weak in the coating, the coating around the pores can be deformed and produced some small cracks under great stress and cutting force. With the crack propagation which will have a strong impact on the structural efficiency of the coating, at last, the coating will peel off.

4 Conclusions

In this paper, the Al_2O_3-13wt% TiO_2 coating is deposited with agglomerated feedstock powders. The AT13 TiO_2 coating is grey, and both γ-Al_2O_3 and α-Al_2O_3 phases are observed in the as-sprayed coatings, the mian phase is γ-Al_2O_3. There are white particulates Al_2O_3 on the surface.

The as-sprayed conventional ceramic coating has a typical lamellar stacking characteristic, and there is no peeling off between the substrate and the coatings. A lot of oxide is existed in the AT13 coating which can improve the microhardness of coating, and the microharness of coating is 1.6 times that of the substrate.

In order to realize the continuous extrusion, the blank entering into the extrusion wheel will need surface friction between the race and blank. Friction coefficient of the coating is a bit bigger than that of the substrate and the abrasion loss of the coating is lower than the substrate, which is only 28% of the substrate. All these can meet the working conditions of the extrusion wheel.

Ata pressure of 25 N, abrasive dusts are not hard enough to resist the crush and broken into tiny particles which can share the load evenly. As a result, chunks of wear debris can not be left on the surface of coatig, and there is no obvious cracks on the surface, it can be seen that the coating has high toughness, which can improve the fatigue property of extrusion wheel, then the extrusion wheel´s life will be prolonged.

Plasma sprayed AT13 coatings on the surface of extrusion wheel is used in the extrusion wheel of TJ700 continuous extrusion machine. The life of the extrusion wheel can be improved 1.2 times, about 330 h.

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