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Friction and Wear Performances of Cathodic Arc Ion Plated TiAlSiN Coating under Oil Lubricated Condition

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Abstract: TiAlSiN coating was deposited on H13 hot work mould steel using cathodic arc ion plating (CAIP). The surface-interface morphologies and phases of the obtained coating were analyzed using field emission scanning electron microscopy (FESEM) and X-ray diffraction (XRD), respectively, and the morphologies, distributions of chemical elements and profiles of worn tracks were also researched using scanning electron microscopy (SEM), energy disperse spectroscopy (EDS), and optical microscope (OM), respectively. The friction-wear performances of TiAlSiN coating under oil lubricated and dry friction conditions were investigated, and the wear mechanisms of TiAlSiN coating were discussed. The experimental results show that the coating is primarily composed of (Ti, Al)N, AlTiN, and TiN hard phases, Si₃N₄ exists between the (Ti, Al)N crystal grains, increasing the coating microhardness to 3200HV. The TiAlSiN coating has excellent performances of reducing friction and wear resistance, the average coefficient of friction (COF) of TiAlSiN coating under oil lubricated condition is only 0.05, lowered than the average COF of 0.211 under dry friction condition, the wear rate decreases by about 81.2% compared with that under dry friction condition. The wear mechanism of TiAlSiN coating under oil lubricated and dry friction conditions is composed of abrasive wear, fatigue wear, and abrasive wear, respectively. The internal friction of oil lubrication is a main factor of decreasing fatigue wear.

Key words: cathodic arc ion plating (CAIP); TiAlSiN coating; coefficient of friction (COF); oil lubrication; dry friction

1 Introduction

In recent years, with the development of coating materials and technologies, their excellent advantages such as wear resistance, corrosion resistance, mechanical properties, and *etc.* have greatly played an important role in the industry^[1,2]. The application fields of TiAlSiN coating are also expanding^[3,4], which have been widely used in the fields of aerospace, automobile, remanufacturing, and *etc.* As one of hot work mould steels, H13 steel is widely used in casting moulds of automobile because of good erosion resistance and high hardened ability, which is one of the fastest consumed and highest required mould steels^[5,6]. H13 hot work mould steel is required to

have a high tempering, strength and wear resistance at high temperature. But the working temperature of hot work mould steel is in the range of 400-800 °C in casting nonferrous metals, and the temperature reaches above 1000 °C in casting ferrous metals under high pressure of 20-120 MPa^[7]. At the same time, the hot work mould steel is also subjected to repeated heating and cooling, as well as the wear and corrosion of metal flow, the main failure modes are thermal wear and thermal fatigue, therefore, the surface properties such as oxidation resistance, anti-adhesion, wear resistance, and *etc.* are required. Because the failure of the mould is mostly produced on the surface, surface modification is a key technique of improving service life of the moulds^[8]. At present, the surface modifications of H13 steel include chemical heat treatment, metallurgy, coating, and *etc.* However, the wear resistance and corrosion resistance are poor, which seriously restricts its applications, and the traditional techniques can not meet the industrial demands. CAIP is a kind of widely used coating technology, and the Ar gas is inlet under high vacuum condition, partial ionization of gas and

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coating materials by glow discharge, and make the ion bombardment target, impact the target material ions, so that it is deposited on the work-piece. Compared with other coating technologies, the CAIP technique has many advantages such as high bonding strength and compact structures, realizing the integration of material forming. The TiAlSiN coating with excellent performance of CAIP technology has a broadly applied prospect^[9-11]. In this work, a TiAlSiN coating was deposited on H13 hot work mould steel using a CAIP. The microstructures and properties of the obtained coating were researched, and the friction and wear behaviors of TiAlSiN coating were investigated under oil lubricated condition, which provided an experimental base for the modification treatment of H13 hot work mould steel.

2 Experimental

H13 hot work mould steel was adopted as the substrate with the chemical compositions as follows (wt%): C 0.32-0.45, Si 0.80-1.20, Mn 0.20-0.50, Cr 4.75-5.50, Mo 1.10-1.75, V 0.80-1.20, P, S \leq 0.030, and the rest was Fe. Before being put into the vacuum chamber, the samples were cleaned using an ultrasonic oscillator for 10min, and dried up. To deposit TiAlSiN coating, industrial high purity N₂ was used as the working gas, and each target of Al and Ti with atom ratio of 50% was adopted. The technological parameters of CAIP are shown as follows: bias power of -100 V, target electric current of 70 A, duty cycle by 30%, gas pressure of 1.2 Pa, working temperature of 500 °C, and deposition time of 120 min. After the samples were pre-treated, the surface-interface morphologies and phases of the obtained coating were analyzed using a JSUPRA55 type FESEM and D/max2500PC type XRD, respectively. The friction and wear tests were conducted on a HSR-2M type reciprocating friction and wear tester under oil (CF-4, 15W/40) lubricated condition, and the COFs of TiAlSiN coating was recorded by a computer, at the same time, the real-time graphics were displayed and the related data were stored. The test parameters were: friction pair: Si₃N₄ ball with the diameter of 5 mm, load of 5 N, wear time of 30 min, frequency of 500 times/min, and wear length of 3 mm. After the wear was finished, profiles of worn track on the TiAlSiN coating were analyzed using a VHX-700FC type three-dimensional optical microscope(OM) with super depth of field, and the morphologies and plane scans of worn tracks were analyzed using a JSM-6360LA type SEM

and its configured EDS, respectively.

3 Results and discussion

3.1 Surface-interface morphologies

Fig.1(a) shows the morphology of TiAlSiN coating. The surface was relatively flat, no obvious defects, showing that CAIP had high surface quality. There were white particles and pits on the surface, which was due to the effect of the anti-sputtering. Although the surface quality of TiAlSiN coating was reduced at a certain degree, the effect on mechanical properties was smaller. Fig.1(b) shows the microstructures of TiAlSiN coating interface, which was closely combined with the substrate, the thickness was about 2 μ m, a dense diffusion layer formed at the interface, improving the bonding strength of TiAlSiN coating interface.

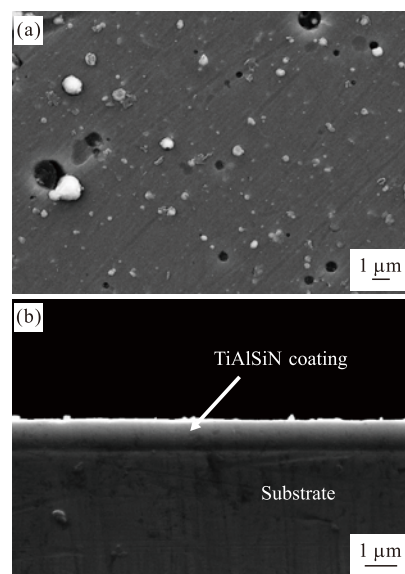


Fig.1 Surface (a) and interface (b) morphologies of TiAlSiN coating

3.2 XRD analysis

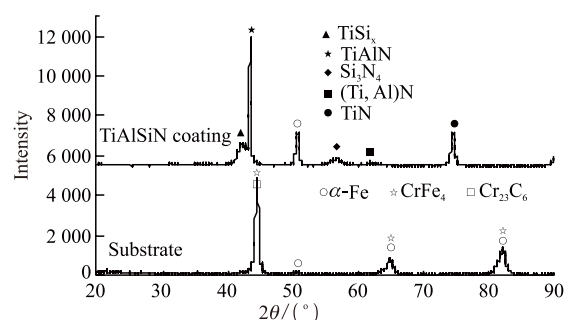


Fig.2 XRD patterns of TiAlSiN coating and substrate

Fig.2 shows the XRD analysis results of TiAlSiN coating and H13 hot work mould steel. The phases of the substrate were composed of α -Fe and CrFe₄. It can

be seen that the TiAlSiN coating was composed of TiSi, TiAlN, TiN, AlTi, (Ti, Al)N, TiN and Si₃N₄ phases. This was because Si₃N₄ was an amorphous phase that was not dissolved in the (Ti, Al)N cell, existing between the (Ti, Al)N crystal grains, to prevent the growth of (Ti, Al)N crystal grains^[12], forming an amorphous structure of Si₃N₄ phase packed (Ti, Al)N. Therefore, the AlN and TiN grains of TiAlSiN coating were refined to form a denser structure, which was conducive to improving the micro-hardness of TiAlSiN coating to 3200HV, which was measured using a JMTT-1000 type micro-hardness tester, increasing 1300HV than that of TiN coating. The addition of Si was a main mechanism of high hardness. This was because the atom radius of Ti was 2 nm, while that of Al was 1.82 nm, the atom radius of Ti was slightly larger than that of Al, the Al atoms were dissolved in the TiN lattice with the replacement method to replace some of Ti atoms in the TiN, and generate TiAlN. After the Ti was replaced, the distortion of crystal lattice was larger, resulting in more than a number of dislocations to reduce the slipping phenomenon, making the micro-hardness of TiAlN phase higher^[13]. The AlN and TiN grains produced a thinning phenomenon, and formed a more compact structure, which was beneficial to improving the micro-hardness of TiAlSiN coating.

3.3 COFs and wear rate

Fig.3 (a) shows that the average COF of TiAlSiN coatings under dry friction condition and oil lubricated conditions is 0.211 and 0.05, respectively, and the wear process was divided into running-in period and stable period. At the running-in period (0-1 min), the COFs of TiAlSiN coating increased rapidly under dry friction condition, and the average COF was 0.174, below the average COF of 0.211. When the wear was continued, the COFs tended to stable state, and entered into the stable period. At the stable period (1-30 min), the surface roughness of TiAlSiN coating was gradually reduced, and the COFs began to decrease, and the average COF was 0.207. The COFs of TiAlSiN coating raised slowly, which was due to the existence of peeling off and wear on the reciprocating zone. Under oil lubricated condition, the COFs of TiAlSiN coating were obviously lowered than those under dry friction condition, and there was no running-in period. The COF of the coating tended to be stable in the friction and wear process, and there was no large fluctuation. The wear profiles of TiAlSiN coating under different conditions are shown in Fig.3(b). The depth of worn track was about 2 μm under dry friction condition

and reached the TiAlSiN coating thickness of 2 μm, indicating that the wear rate was $133.3 \times 10^{-10} \text{ mm}^3/\text{N}\cdot\text{s}$ and the TiAlSiN coating was worn out. Under oil lubricated condition, the depth of worn track was about 0.5 μm, and the wear rate was only $25 \times 10^{-10} \text{ mm}^3/\text{N}\cdot\text{s}$, decreased by about 81.2% compared with that under dry friction condition. The depth of worn track was less than the TiAlSiN coating thickness of 2 μm, indicating that the TiAlSiN coating was not worn out and the wear was carried in the TiAlSiN coating. As a result, the TiAlSiN coating had excellent property of reducing friction and wear resistance under oil lubricated condition.

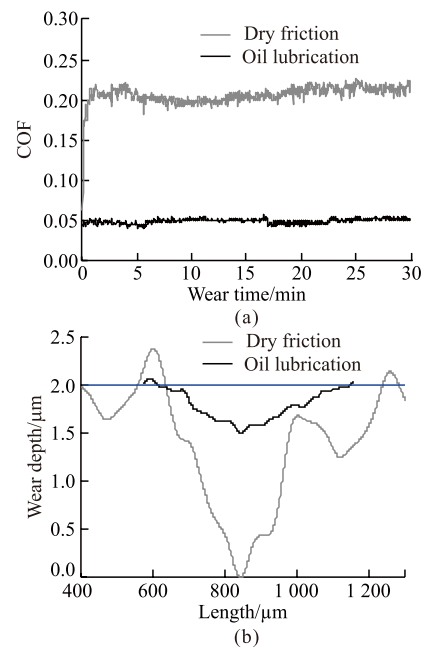


Fig.3 COFs vs wear time (a) and wear profiles of worn tracks (b)

3.4 Plane scans of worn tracks and EDS analysis

Fig.4(a) shows the worn track of TiAlSiN coating under dry friction condition for 30 min, the width of worn track was about 900 μm, there were many obvious furrows with serious wear, no cracking and peeling phenomenon. The plane scan result of worn track is shown in Fig.4 (b), and the mass fractions (mass, %) were as follows: Ti 31.55, Al 11.57, Si 5.95, Fe 47.71, and Cr 3.20. The Ti, Al, and Si were derived from the TiAlSiN coating, while the Cr and Fe came from the H13 hot work mould steel. The contents of Ti and Al on the worn track were less, as shown in Figs.4 (c) and 4(d), which showed that the nitrides of Ti and Al were almost worn out in the wear test. Si on the worn track was uniformly distributed, as shown in Fig.4(e), and there was no accumulation phenomenon, which was mainly because the substrate also contained Si. Fe and

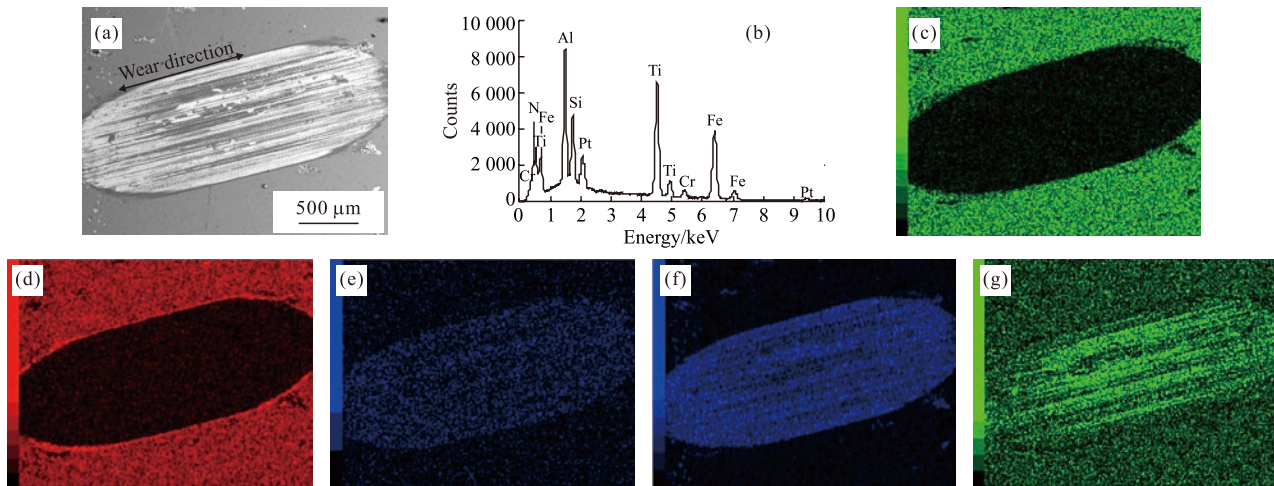


Fig.4 Plane scan analysis of TiAlSiN coating under dry friction condition: position of plane scan analysis (a), result of plane scan analysis (b), Ti content (c), Al content (d), Si content (e), Fe content (f), and Cr content (g)

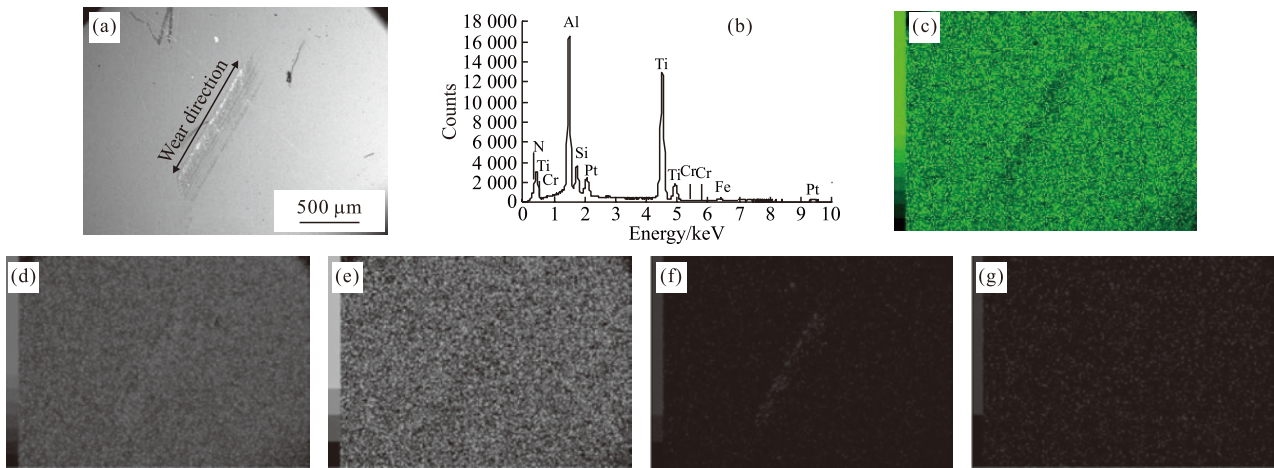


Fig.5 Plane scan analysis of TiAlSiN coating under oil lubricated condition: position of plane scan analysis (a), result of plane scan analysis (b), Ti content (c), Al content (d), Si content (e), Fe content (f), and Cr content (g)

Cr of the substrate produced the atoms-rich zones on the worn track, as shown in Figs.4(f) and 4(g), which indicated that the TiAlSiN coating was worn out.

Fig.5(a) shows the worn track of TiAlSiN coating under oil lubricated condition for 30 min. The width of worn track was 400 μm , decreased by about 55.6%, compared with that under dry friction condition, and the depth of worn track was relatively shallow. The TiAlSiN coating did not show obvious peeling off. The plane scan result of worn track is shown in Fig.5(b), and the mass fractions (mass, %) were as follows: Ti 70.33, Al 20.97, Si 4.44, Fe 3.87, and Cr 0.39. Among them, Ti, Al, and Si were derived from the TiAlSiN coating, while Cr and Fe were derived from H13 hot work mould steel. From Figs.6(c) and 6(d), it can be seen that the Ti and Al on the worn track were uniformly distributed, which indicated that the nitrides of Ti and Al were not worn away in the wear test. Si on the worn track was uniformly distributed, as shown in Fig.5(e), and there was no accumulation phenomenon,

which was mainly because the substrate also contained Si. Fe and Cr on the worn track were also uniformly distributed, as shown in Figs.5(f) and 5(g), and there was no accumulation phenomenon, which indicated that the TiAlSiN coating was not worn out.

3.5 Mechanism analysis

Under dry friction condition, the TiAlSiN coating surface was badly worn, the worn debris was more, the wear mechanisms was abrasive wear, as shown in Fig.6(a). This was because the TiAlSiN coating was worn out, the TiAlSiN coating hardness was relatively higher, the rough peaks of the friction pair were embedded into the TiAlSiN coating, and ploughed into many grooves. As shown in Fig.6(b), the worn surface was rough, there were obvious peelings and peeled pits, showing a more serious abrasive wear and characteristics of fatigue wear, and the reason was mainly that the TiAlSiN coating surface displayed plastic deformation, causing proliferation and movement of dislocation. The dislocation pile-

up formed along the plastic deformation direction, the crack source was initiated in the surface layer, and the crack led to fracturing and peeling^[14]. Therefore, the surface roughness increased, resulting in increasing of the COFs in Fig.3(a).

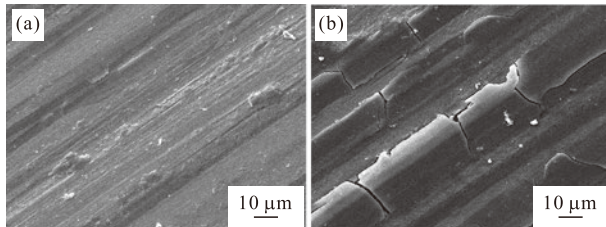


Fig.6 Morphologies of worn scar under dry friction condition: abrasive wear (a), and peeling off (b)

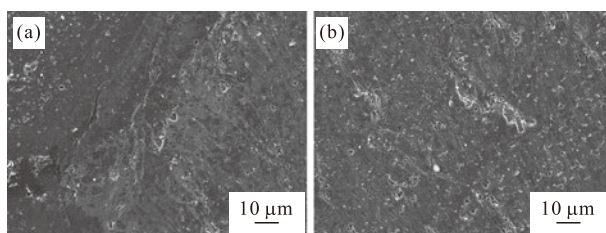


Fig.7 Morphologies of worn track under oil lubricated condition: abrasive wear (a), and cracking (b)

Under oil lubricated condition, the wear mechanism of TiAlSiN coating was abrasive wear, and the tendency of peeling decreased, as shown in Fig.7(a). This was because the worn track was shallower and the TiAlSiN coating was not worn out, which indicated that oil lubrication reduced the peeling tendency of TiAlSiN coating. However, there was a large number of worn debris. The main reason was that the friction resistance was determined by the internal friction of lubrication oil in the process of fluid lubrication. Generally, the higher the oil viscosity was, the lower the COF was, and the more obvious the effect of friction reduction was^[15]. Because the reciprocating test resulted in increased friction temperature, the viscosity and thickness of oil film decreased, which caused the shearing stress concentration on the micro convex body of TiAlSiN coating, and the actually contacted area between the TiAlSiN coating and the ceramic ball increased, so the wear increased, and a large number of fine worn marks and cracks appeared on the TiAlSiN coating surface, as shown in Fig.7(b), but the peeling of the TiAlSiN coating was not found.

4 Conclusions

a) The TiAlSiN coating is primarily composed of (Ti, Al) N, AlN, and TiN phases, the Si content

increases its micro-hardness to 3200HV.

b) The average COF of TiAlSiN coating under oil lubricated condition is 0.05, obviously lower than that of 0.211 under dry friction condition, and the wear rate decreases by 81.2%, showing that the TiAlSiN coating has an excellent friction reduction and wear resistance.

c) The wear mechanism of TiAlSiN coating under dry friction condition is abrasive wear and fatigue wear, while that under the oil lubricated condition is abrasive wear, and the wear resistance is determined by the internal friction of lubrication oil.

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