MICROSTRUCTURE AND TRIBOLOGICAL BEHAVIOR OF W-Mo ALLOY COATING ON POWDER METALLURGY GEARS BASED ON DOUBLE GLOW PLASMA SURFACE ALLOYING TECHNOLOGY

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Abstract

In the present paper, plasma surface alloying was implemented on powder metallurgy gears to improve its wear resistance based on double glow plasma surface metallurgy technology. A W-Mo alloy coating was obtained in the process. The morphology, microstructure and phase composition were investigated by SEM, EDS and XRD. The hardness was examined by Vickers hardness test and nanoindentation test. The tribological behavior of powder metallurgy gears before and after plasma surface alloying was evaluated on a ball-on-disc reciprocating sliding tribometer under dry sliding condition at room temperature. The results indicate that the W-Mo alloy coating is homogeneous without defects, which includes deposition layer and interdiffusion layer. The average microhardness of powder metallurgy gears before and after plasma surface alloying is 145.8 $HV_{0.1}$ and 344.4 $HV_{0.1}$, respectively; Nano hardness of deposition layer and interdiffusion layer is 5.76 GPa, 14.35 GPa, respectively. The specific wear rate of W-Mo alloy coating is lower than original PM gears. The wear mechanism of W-Mo alloy coating is slight adhesive wear. The W-Mo alloy coating prepared by double glow plasma surface alloying technology can effectively improve wear resistance of powder metallurgy gears.

Keywords: Plasma surface alloying; Microstructure; Friction and wear; Wear mechanism; Powder metallurgy gears

1. Introduction

Powder metallurgy (PM) plays a major role in many fields as an advanced manufacturing technology, and the main attraction of PM materials is that it exhibits lots of excellent properties such as good thermal conductivity, machine ability along with economic performance [1-4]. However, the finite wear resistance of traditional iron-based PM materials limits their application in harsh working condition. S.B. Halesh et.al [5] studied wear behavior of powder metallurgy Fe15Al15Cu materials under lubricated and unlubricated sliding conditions. The result shows that the powder metallurgy Fe15Al15Cu materials have poor wear resistance at 10 kg normal load.

Techniques such as warm compaction [6], high speed compaction [7-8] and die wall lubrication [9] can improve wear resistance of PM materials. However, those methods would not only have sophisticated craft but also result in expensive cost.

Nowadays, surface strengthening methods, laser cladding, surface densification, and ion nitridation, have been introduced to improve the wear resistance of materials. M. Keddam et.al [10] thought that plasma nitriding is one of the widely used surface treatments methods for improving the surface wear resistance of materials. Sirong Yu et.al [11] indicated that iron-based powder coatings containing nano-Al_2O_3 particles prepared by laser cladding improved the wear resistance of iron-based powder metallurgy materials. Good metallurgical bond was formed between laser cladding zone and substrate zone. However, the temperature of laser cladding is usually high, which has possibly a negative effect on other

performance of PM materials. Teruie Takemasu et.al [12] carried out surface rolling on PM gears and studied the effect of surface rolling on wear resistance of PM gears. Surface densification treatment was performed [13-15] on PM gears and the effect of dense layer on wear resistance was studied. Some scholars focused on improving wear resistance of PM parts by ion nitridation [16-17].

Double glow plasma surface alloying technology is an innovative technology developed based on ion nitriding technology. The advantage of double glow technology is that it can improve the wear resistance, hardness and oxidation resistance of metal or alloy [18-20]. An alloy coating prepared by double glow plasma surface alloying technology consists of two part: deposition layer and interdiffusion layer. The deposition layer displays loose structure due to sputtering, which has effect on the wear mechanism. The alloy coating is metallurgically bonded with substrate due to presence of interdiffusion layer and the bonding strength is high. Alloy elements chromium, nickel, tantalum, aluminum and their combinations have been already successfully applied on metal or alloy by double glow plasma surface alloying at present [21-23]. However, there are few reports about application of double glow plasma surface alloying technology on iron-based PM materials.

The metals W and Mo, both with body-centered cubic structures, which have high melting point and good thermodynamic stability, high hardness, and modulus of elasticity, are beneficial for improving wear resistance. However, addition of W and Mo in sintering process may affect other properties such as fatigue of powder metallurgy materials. Double glow plasma surface alloying technology, which has good economic performance and has no negative effect on other properties of substrate, provides a new pathway for improving wear resistance of powder metallurgy materials.

Therefore, W-Mo alloy coatings were prepared on iron-based PM gears by double glow plasma surface alloying technology in this paper. The morphology, microstructure and phase composition were investigated by SEM, EDS and XRD. The hardness was examined by Vickers hardness test and nanoindentation test. Tribological behavior of the alloy coating under different sliding speeds was investigated by CET-I ball-on-disc reciprocating sliding tribometer at room temperature.

2 Experimental details
2.1 Materials and methods
The chemical composition of iron-based powder metallurgy gears was 1.7-1.95 Ni, 1.1-1.3 Cu, 0.4-0.6 Mo, <0.03 P, <0.02 S, <0.02 O, and balance Fe

(wt.%). The W-Mo alloy target, prepared by the powder metallurgy method, was cylindrical (φ100 mm, d5 mm), and the element ratio was W20-Mo80 (wt.%). The original PM gears were ground with sandpaper and polished to a roughness Ra<0.1 μm. Prior to the preparation of W-Mo alloy coating, all samples and W-Mo alloy target were cleaned for 20 minutes by methanol in an ultrasonic cleaner.

Process parameters of plasma surface alloying are shown in Table 1. Schematic diagram of double glow plasma surface alloying equipment is shown in the Fig 1. The double glow refers to two glows in vacuum chamber, one of which happened between the anode and the cathode, the other one happened between the anode and the source electrode. The ions, sputtered from W-Mo target, were bombarded toward PM gears and W-Mo alloy coating was formed.

Table 1. *Process parameters of double glow plasma surface alloying*

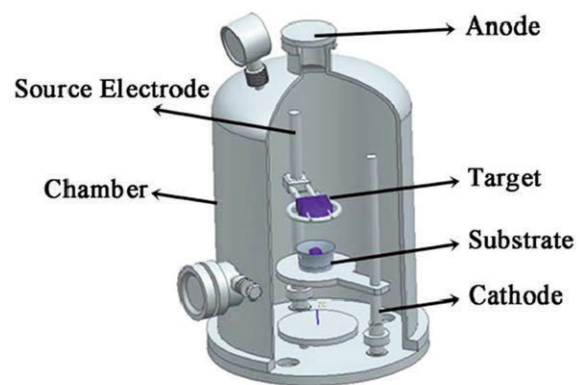| Parameters	Settings
Temperature /K	1123
Time/h	3
Working pressure/Pa	36~42
Distance between the source and substrate/mm	16~22
Voltage of the source/V	830~960
Voltage of the substrate/V	470~570

Figure 1. *Schematic diagram of double glow plasma surface alloying equipment*

2.2 Microstructure analysis

The surface morphology, cross-sectional morphology, and chemical composition of PM gears before and after plasma surface alloying were characterized by a scanning electron microscope (SEM) that was equipped with energy-dispersive X-ray spectrometer (EDS). The phase composition of W-Mo alloy coating was performed by X-ray diffraction

(XRD) with CuKα radiation over arrange of 2θ from 10° to 90°.

2.3 Microhardness test

The microhardness of PM gears before and after plasma surface alloying was performed by HXS-100A Vickers hardness tester. Five points were taken on surface of samples and the average values with standard deviation were calculated.

2.4 Nanoindentation test

The characteristic of nanoindentation test is that it can avoid interaction influence of different zones due to press-in depth. The nanoindentation test of deposition layer and interdiffusion layer was carried out with Shimadzu dynamic nanoindentation tester (DUH-W201) equipped with a Berkovich diamond tip nano-indenter. The nanoindentation test was executed by driving the indenter at a constant loading rate of 0.7553 mN/s into surface of W-Mo alloy coating with the maximum applied load of 100 mN. In addition, deposition layer was eliminated by polishing before nanoindentation test for interdiffusion layer.

2.5 Friction and wear test

The PM gears before and after plasma surface alloying were subjected to friction and wear test using CET-I ball-on-disc reciprocating sliding tribometer under different sliding speeds (300 rpm, 400 rpm, 500 rpm) at room temperature. After previous experimental verification, the degree of wear on substrate and alloy coating was insufficient when load was too small. When the load was too large, alloy coating was worn away in a short time. Therefore, wear test was performed at 470 g normal load. The antagonistic ball was made of Si_3N_4, which diameter was 4.5 mm. The surface profile was characterized by surface profiler. Wear volumes and specific wear rates were calculated. Further, specific wear rates were calculated based on the formula presented below [24]:

$$K = V/PS \qquad (1)$$

where K was the specific wear rate ($mm^3 N^{-1} m^{-1}$), V was the wear volume (mm^3), P was the load (N) and S was the total sliding distance (m).

3. Results and discussion
3.1 Microstructure and phase composition of W-Mo alloy coating

Figure 2 shows the surface morphology and chemical composition of W-Mo alloy coating prepared by plasma surface alloying. The W-Mo alloy coating was homogeneous without defects such as porosity and crack and there were cellular bumps on the surface. EDS result shows that the surface consisted of W, Mo, and Fe. The chamber and fixture of double glow plasma surface alloying equipment in Figure 1 were made of steel. The Fe ions in steel are inevitably involved in sputtering during experiment. Therefore, EDS result shows that the surface consisted of W, Mo, and Fe.
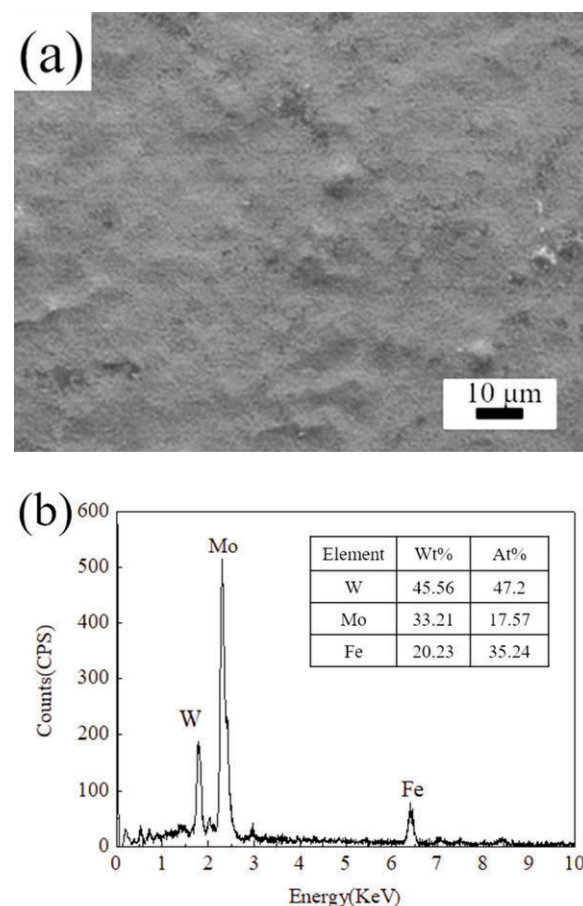

Figure 2. Surface morphology and EDS analysis of W-Mo alloy coating: (a) surface morphology (b) chemical composition

Figure 3 shows the cross-section morphology and the chemical composition distribution of W-Mo alloy coating prepared by double glow plasma surface alloying. The W-Mo alloy coating combined well with substrate and included two part, as shown in Figure 4(a). Zone I and II were W-Mo sputtering deposition layer and interdiffusion layer, respectively. The overall thickness of W-Mo alloy coating was about 15 μm and the thickness of deposition layer and interdiffusion was about 11 μm and 4 μm, respectively. There is no obvious boundary between interdiffusion layer and substrate, which improves

bonding force. The interdiffusion layer consists of Fe from the PM gears diffuses into the coating and W-Mo from coating diffuses into the PM gears. In interdiffusion layer, the elements W and Mo decrease gradually and the Fe increases gradually.
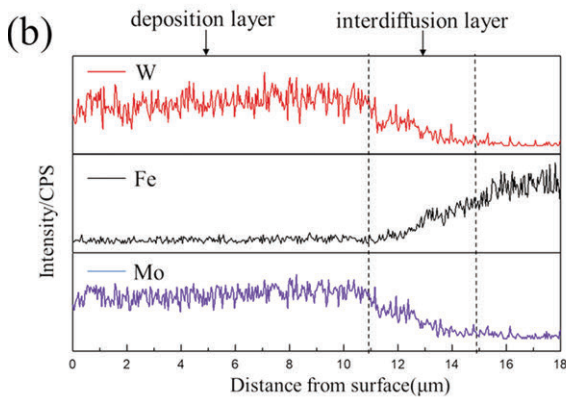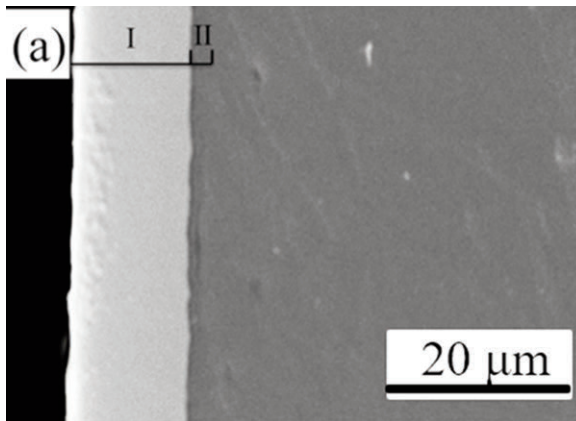

Figure 3. Cross-sectional morphology and EDS analysis of W-Mo alloy coating: (a) cross-sectional morphology (b) component distribution

Figure 4 shows XRD spectrum of W-Mo alloy coating. As we can see, the W-Mo alloy coating consisted of pure W, pure Mo, and intermetallic compound Fe_3Mo. W and Mo phases had a high melting point, hardness, and strength, therefore, their occurrence may strengthen W-Mo alloy coating. The intermetallic compound Fe_3Mo in interdiffusion layer can increase the resistance of plastic deformation.

3.2 Mechanical behavior

The microhardness of original PM gears was about 136.5~152.2 $HV_{0.1}$, and average value was 145.8 $HV_{0.1}$. The microhardness of W-Mo alloy coating was about 310.6~370.6 $HV_{0.1}$, and average value was 344.4 $HV_{0.1}$. The standard deviation of average hardness was 43.35, which indicated the W-Mo alloy coating was homogeneous. The microhardness of W-Mo alloy coating prepared by
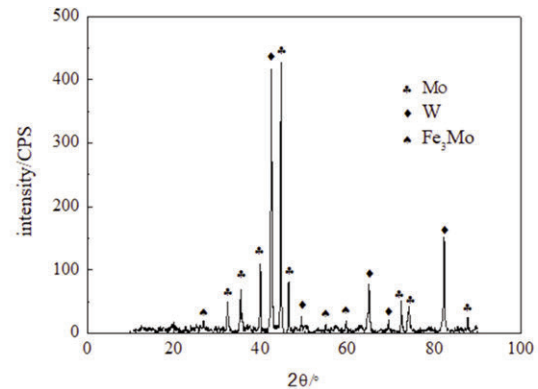

Figure 4. XRD spectrum of W-Mo alloy coating

plasma surface alloying was significantly higher than in original PM gears.

Figure 5 displays force-depth curves under 100 mN load for deposition layer and interdiffusion layer of PM gears after double glow plasma surface alloying. The maximum indentation depth of deposition layer is 0.814 μm and the residual indentation depth is 0.605 μm. The maximum indentation depth of interdiffusion layer is 0.515 μm and the residual indentation depth is 0.317 μm, which is obviously lower than deposition layer. According to speculation, the interdiffusion layer mainly consisted of intermetallic compounds and supersaturated solid solution, which increased the resistance of plastic deformation [25]. XRD, shown in Figure 4,indicates that intermetallic compound Fe_3Mo is present in interdiffusion layer and it may increase the resistance of plastic deformation. Figure 6 shows hardness and elastic modulus of deposition layer and interdiffusion layer. The hardness of deposition layer was 5.76 GPa, and elastic modulus was 154 GPa. In contrast, the hardness of interdiffusion layer was 14.35 GPa, and the elastic modulus was 221 GPa. It indicates that the interdiffusion layer possesses better elastic recovery ability and lower plastic deformation than the deposition layer.
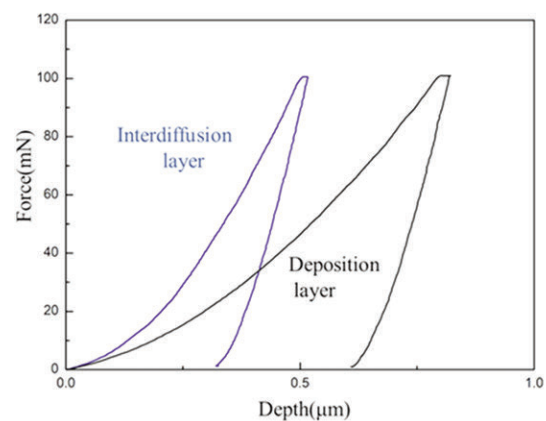

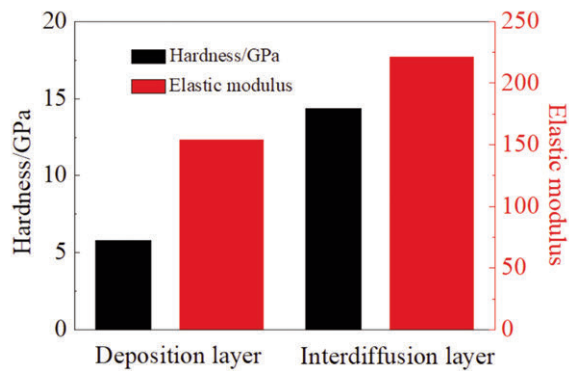*Figure 5.* Force-depth curves under 100 mN load for interdiffusion layer and deposition layer

Figure 6. *The hardness and elastic modulus of deposition layer and interdiffusion layer*

3.3 Friction and wear properties

The variations in friction coefficient of PM gears before and after plasma surface alloying under different sliding speeds are shown in Figure 7. Firstly, the friction coefficient of original PM gears was about 0.6~0.7, and that of the W-Mo alloy coating prepared by plasma surface alloying was about 0.3~0.45. It is noted that the W-Mo alloy coating exhibits significant lower friction coefficient than the original PM gears under different sliding speeds. Secondly, it reveals that friction coefficient of the W-Mo alloy coating exhibits less fluctuating than that of original PM gears under different sliding speeds.

For original PM gears, it is easier to generate abrasive particles on the rough surface than W-Mo alloy coating surface under a load. These abrasive particles on the surface are easily embedded into original PM gears, which cause serious wear and fluctuating of friction coefficient. The deposition layer of W-Mo alloy coating is loose due to sputtering and has well performance of plastic deformation. The deposition layer plays a role in reducing friction and lubricating as soft film during the friction. Therefore, W-Mo alloy coating shows lower friction coefficient than original PM gears. In addition, due to high hardness of the W-Mo coating prepared by plasma surface alloying, abrasive particles are embedded difficultly into the surface. The friction coefficient curves of the W-Mo alloy coating become stable as particles peeled off.

Different sliding speeds have little significant effect on friction coefficient of the W-Mo alloy coating. But friction heat is generated during friction. The effect of friction heat is strengthened with increase of sliding speed, caused the friction coefficient change. In W-Mo alloy coating, abrasive particles were generated by wear and the friction coefficient increased when the sliding speed was 300 rpm. When the sliding speed increased, the surface generated a film under influence of friction heat,

which consisted of abrasive particles and had lubrication effect. Therefore, the friction coefficient decreased to some extent.

Figure 8 shows the surface profiles of wear tracks of PM gears before and after plasma surface W-Mo alloying under different sliding speeds. Table 2 shows wear volume and specific wear rate under different sliding speeds. As sliding rates increased, the increment degree of width, depth and wear volume of
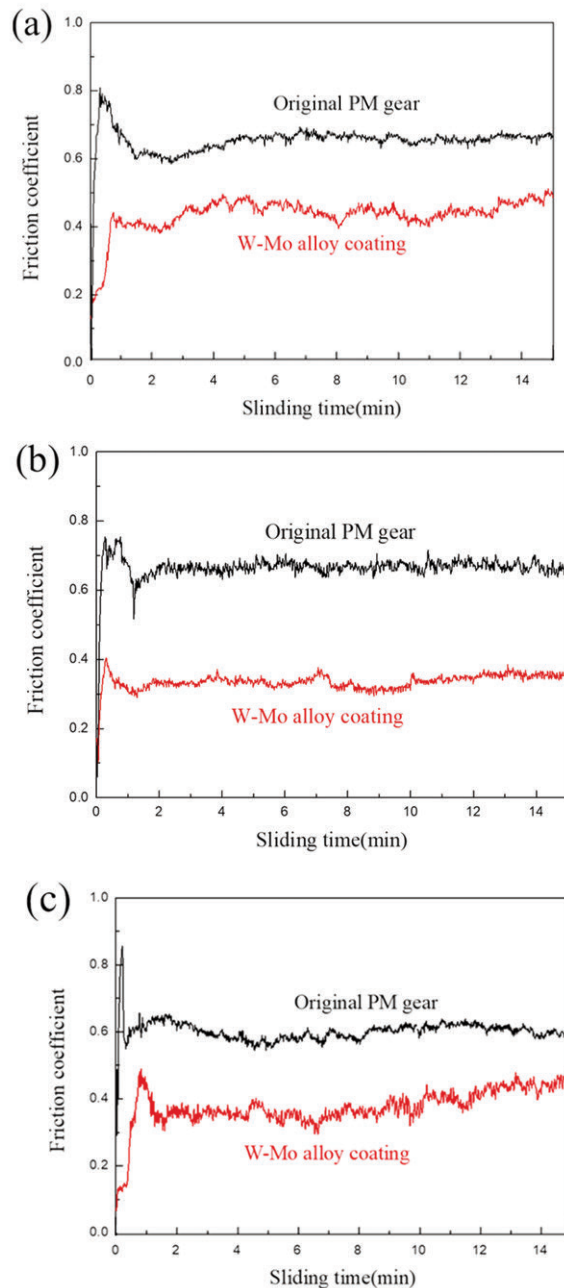

Figure 7. *Friction coefficient curves of PM gears before and after plasma surface alloying under different sliding speeds (a) 300 rpm (b) 400 rpm (c) 500 rpm*
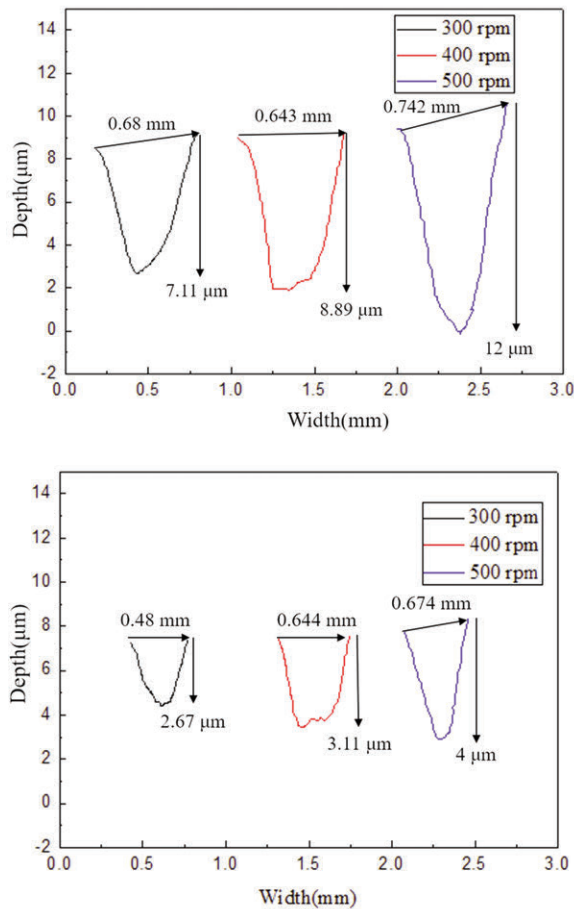
Figure 8. *Surface profiles of wear tracks under different speeds (a) original PM gears(b) PM gears after plasma surface alloying*

W-Mo alloy coating was significantly lower than those of original PM gears. The specific wear rate of W-Mo alloy coating was about 25~35% of original PM gears under different sliding speeds, which indicates that W-Mo alloy coating prepared by double glow plasma surface alloying technology improves wear resistance of original PM gears.

Figure 9, 10, and 11 show the wear surface morphology of PM gears before and after plasma

Table 2. *Wear volume and specific wear rate of PM gears before and after plasma surface alloy coating under different sliding speeds*

Materials	Sliding velocity (r/min)	wear volume ($\times 10^{-3}$/mm³)	specific wear rate ($\times 10^{-3}$mm³/N⁻¹m⁻¹)
Original PM gears	300	16.12	0.699
	400	19.05	0.827
	500	29.68	1.289
W-Mo alloy coating	300	4.27	0.185
	400	6.68	0.290
	500	8.98	0.389

surface alloying under sliding speeds of 300 rpm, 400 rpm, and 500 rpm, respectively. Grooves and wear debris appear on surface of original PM gears under
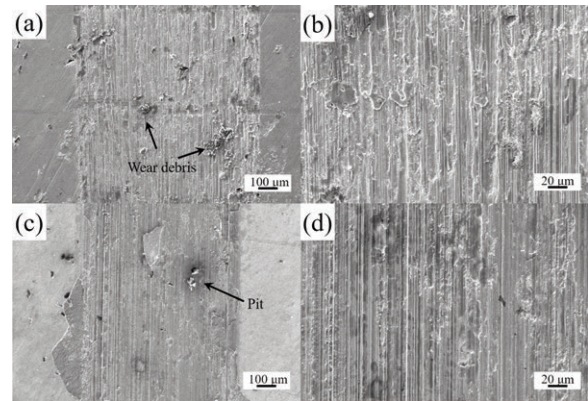

Figure 9. *Wear surface morphology of PM gears before and after plasma surface alloy coating under sliding speed of 300 rpm (a) (b) original PM gears (c) (d) W-Mo alloy coating*
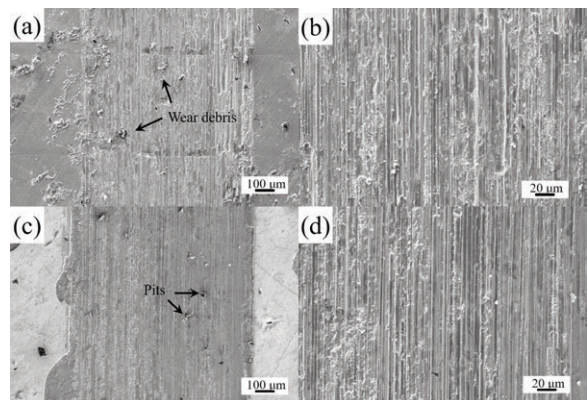

Figure 10. *Wear surface morphology of PM gears before and after plasma surface alloy coating under sliding speed of 400 rpm (a) (b) original PM gears (c) (d) W-Mo alloy coating*

Figure 11. *Wear surface morphology of PM gears before and after plasma surface alloy coating under sliding speed of 500 rpm (a) (b) original PM gears (c) (d) W-Mo alloy coating*
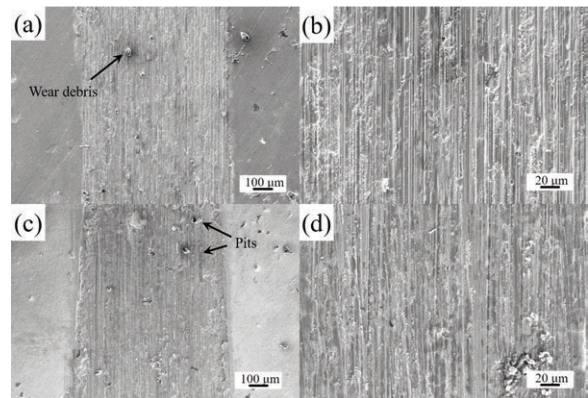
different sliding speeds, indicating that the wear mechanism of original PM gears is abrasive wear. Moreover, the grooves become deeper as the sliding speed increase. Due to poor hardness and plastic deformation capacity of the original PM gears, abrasive debris was subjected to plough under a load, which resulted in many parallel grooves. Abrasive particles were easily embedded into the surface of original PM gears under high sliding speed, resulting in deeper grooves.

The W-Mo alloy coating prepared by plasma surface alloying displayed wear surface morphologies different from the original PM gears. There are a few fine pits and slight grooves which appear on the surface of W-Mo alloy coating, only less or no wear debris is generated. It is worth noting that the wear depth of W-Mo alloy coating was lower than thickness of deposition layer, which manifest wear process and destruction that happened in deposition layer. In addition, the deposition layer has not peeled off from PM gears. The deposition layer of W-Mo alloy coating was loose due to sputtering and had well performance of plastic deformation. The deposition layer played a role in reducing friction and lubricating as soft film during the friction, which reduced the impact of ploughing action, and left only slight grooves. In the process of wear, the cellular bumps on surface of W-Mo alloy coating were plastically deformed along sliding direction and local area of W-Mo alloy coating was welded with the friction pair. The local welding area on soft surface of deposition layer was torn, resulted in pits. The wear mechanism of W-Mo alloy coating is slight adhesion wear. The gradient distribution structure and good adhesion between deposition layer and interdiffusion layer prevents deposition layer peeling off.

In summary, from the analysis of friction coefficient, specific rate and wear surface morphology, it can be concluded that under the conditions of different sliding speeds, the W-Mo alloy coating prepared by double-glow plasma surface alloying technique can effectively improve wear resistance of the original PM gears.

4. Conclusions

The PM gears before and after double glow plasma surface alloying technology, was investigated to evaluate the wear resistance. The conclusions derived from this paper are summarized as follows:

(1) W-Mo alloy coating prepared by double glow plasma surface alloy coating includes deposition layer and interdiffusion layer. The W-Mo alloy coating is homogeneous without defects.

(2) W-Mo alloy coating improves microhardness of original PM gears. Interdiffusion layer possesses better elastic recovery ability and lower plastic deformation than deposition layer. According to speculation, the intermetallic compound Fe_3Mo in interdiffusion layer increases resistance of plastic deformation.

(3) Deposition layer plays a role in reducing the friction and lubricating as soft film under the friction and wear process. The wear mechanism of W-Mo alloy layer is slight adhesive wear. The double glow plasma surface alloying technology provides a new pathway for improving wear resistance of the powder metallurgy materials.

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MIKROSTRUKTURA I TRIBOLOŠKE OSOBINE PREVLAKE OD W–Mo LEGURE NA ZUPČANICIMA PROIZVEDENIM POSTUPKOM METALURGIJE PRAHA PO TEHNOLOGIJI LEGIRANJA POVRŠINE DUPLIM MLAZOM PLAZME

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Apstrakt

U ovom radu je predstavljena primena postupka legiranja površine plazmom, na zupčanike dobijene postupkom metalurgije praha u cilju povećanja njihove otpornosti na habanje. Tokom postuka je dobijena prevlaka W-Mo legure. SEM, EDS i XRD analize su korišćene za ispitivanje morfologije, mikrostrukture i sastav faze. Test tvrdoće po Vickers-u i test nano-ozubljenja su korišćeni za ispitivanje tvrdoće materijala. Tribološke osobine ove opreme, pre i posle postupka legiranja površine plazmom, određene su naizmenično kliznim tribometrom sa kuglom na disku, u suvim uslovima na sobnoj temperaturi. Rezultati pokazuju da je prevlaka W-Mo legure homogena i bez defekata, i da se sastoji od nataloženog sloja i difuzionog sloja. Prosečna mikrotvrdoća pre i posle postupka legiranja, iznosi 145.8 $HV_{0.1}$ i 344.4 $HV_{0.1}$. Nanotvrdoća nataloženog sloja iznosi 5,76 GPa, a difuznog sloja 14.35 GPa. Specifična brzina habanja prevlake je manja od početne brzine habanja dela. Mehanizam habanja W-Mo prevlake predstavlja neznatno adhezivno habanje. W-Mo prevlaka dobijena tehnikom legiranja površine duplim mlazom plazme može znatno da poveća otpornost habanja zupčanika dobijenih postupkom metalurgije praha.

Ključne reči: *Legiranje površine plazmom; Mikrostruktura; Trenje i habanje; Mehanizam habanja; Zupčanici dobijena postupkom metalurgije praha*