

Article

Investigation of the Surface Properties and Wear Properties of AISI H11 Steel Treated by Auxiliary Heating Plasma Nitriding

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Abstract: This paper presents an auxiliary heating method to maintain a uniform specimen temperature and precisely control nitriding temperature during plasma nitriding. The surface properties and wear properties of AISI H11 steel treated by auxiliary heating plasma nitriding are investigated. Firstly, the specimens with different diffusion layers and different hardness levels are fabricated through changing the plasma nitriding temperature. Secondly, the surface properties of the plasma-nitrided H11 steel specimens are characterized by a scanning electron microscope (SEM), X-ray diffractometer, metallographic microscope and microhardness tester. The results show that the surface hardness of the plasma-nitrided specimen is almost twice as high as that of the untreated specimen. The thickness of diffusion layer increases with the increase of nitriding temperature. However, the surface hardness firstly increases and then decreases with the increase of the nitriding temperature. Finally, the wear properties of untreated and plasma-nitrided H11 steel specimens are investigated under different friction conditions. The results show that the plasma-nitriding method can significantly improve the wear resistance of AISI H11 steel. The friction coefficient fluctuations of the plasma-nitrided specimens are all lower than those of the untreated specimens. In addition, the wear rates of the plasma-nitrided specimens rise along with load, and reduce along with the sliding speed and friction temperature.

Keywords: AISI H11 steel; auxiliary heating plasma nitriding; surface properties; wear properties

1. Introduction

Hot working die steel plays a vital role in the die industry due to its superior manufacturing and processing performance, and is especially suitable for metal's thermo-plastic forming process, such as hot forging die, die casting die, hot heading die, and so on [1]. In the practical process of hot working die steel, die steel not only bears the friction and wear affect from blank deformation, but also suffers the grinding effect from the oxidized iron filings in high-temperature environments. In addition, thermal fatigue failure and wear failure are the common failure modes of hot working die steel [2,3]. Hence, hardness and wear resistance are two of the key performance indicators of hot working die steel. With the increase of the use requirements of hot working die steel, improving its hardness and wear resistance is always a research hotspot.

AISI H11 steel is one of the widely used hot working die steels which contains Cr, Mo and other alloying elements. Alloy nitrides formed by nitriding treatment have the characteristics of

high hardness, good dispersive distribution and high thermal stability. Hence, nitriding treatment is an alternative surface strengthening treatment method for AISI H11 steel, which can significantly improve its wear resistance, corrosion resistance and fatigue resistance [4]. Nitriding treatment mainly includes gas nitriding, carbonitriding and plasma nitriding [5–7]. Plasma nitriding has many advantages, such as good wear resistance, high diffusion speed, ease to control, little deformation and environment-friendliness [8,9], and is an important surface strengthening method in mold and friction parts. In addition, understanding the wear properties of specimens treated by plasma nitriding under different friction conditions is vital to the mechanical structure design and actual use of the friction parts.

1.1. Plasma Nitriding Treatment

A series of studies about the surface strengthening treatment for hot working tool steel by plasma nitriding had been carried out. Rad et al. [10] applied the method of plasma nitriding to strengthen AISI H11 steel. It could be found that the specimens treated by plasma nitriding had better wear resistance than untreated specimens. Among the plasma-nitrided specimens, the specimen that contained more of the ϵ -Fe₂₋₃N phase exhibited the higher hardness and the lower wear rate. Liu et al. [11] investigated the influences of processing time and temperature on the wear properties of H11 steel treated by plasma nitriding. It was pointed out that the wear rate of H11 steel firstly decreased and then increased with the increase of processing temperature, and decreased with the increase of processing time. Miyamoto et al. [12] and Das et al. [13] proposed a new method for nitriding H13 steel based on neutral nitrogen and nitrogen ions through electron-beam-excited plasma. The diffusion nitriding surface without composite layer was produced by neutral nitriding treatment by the means of precisely controlling the treatment time. This method could effectively improve the wear resistance of H13 steel without altering its surface topography. Zlatanovic et al. [14] presented the hybrid method of plasma nitriding and carburizing after plasma nitriding treatment. This method could further increase the wear resistance, fatigue resistance and corrosion resistance of H11 steel. Jacobsen et al. [15], Karamis et al. [16] and Cruz et al. [17] analyzed the effects of plasma current density and processing temperature on the phase formation and tribological properties of H13 steel by plasma nitriding. It was observed that several phases were formed under the different combinations of plasma current density and process temperature which included the hexagonal ϵ -Fe_{2-3.2}N, cubic γ' -Fe₄N and tetragonal α' -Fe. The ϵ -Fe_{2-3.2}N with hexagonal close-packed (hcp) crystalline structure showed higher hardness than the γ' -Fe₄N phase. The specimen with the higher hardness showed the better wear properties [10].

From the above research, it was pointed out that plasma-nitriding treatment can enhance the hardness and wearability of hot working die steel. However, the cool wall furnace is usually used in the traditional plasma nitriding treatment. One or two thermal insulating layers cover the internal wall of furnace. Then, the temperature of thermal insulating layer is about 400–550 K. The temperature difference between specimen and thermal insulating layer is about 350–400 K. In addition, the decomposition of ammonia gas can absorb a lot of heat energy. This fact can cause imprecise temperature control and uneven temperature distribution on the specimen, in which case the nitriding quality of the specimen will be uncontrollable and nonuniform.

1.2. Wear Properties

Jiang et al. [18] and Leite et al. [19] carried out a set of dry sliding friction tests for H13 steel with different hardness levels under various sliding speeds and loads. It was obtained that the wear resistance had a positive correlation with hardness at relatively low sliding speeds. Karamis et al. [20] investigated the influences of friction conditions on the sliding wear performance of H11 steel treated by plasma nitriding. It was pointed out that the wear mechanism of specimens varied with the load and slip-ratio. Sam et al. [21] made a group of friction tests for H11 steel under various loads at 773 K on a pin-on-disk wear tester. It was found that wear rate first increased, then decreased and then increased with the increase of load. In their study, it could be observed that: when the load was

lower than 30 N, the wear rate increased along with load because of the fragmentation of unstable surface layers and larger fluctuations in the friction coefficient at this load. When the load ranged from 30 N–40 N, the friction coefficient was relatively stable, and a smooth “glaze” surface was formed as a protective layer. Then, the wear rate decreased along with the load. When the load was higher than 40 N, the smooth “glaze” surface was destroyed because of high friction stress, and then the wear rate increased with the increase of the load. Chen et al. [22], Kumar et al. [23] and Zhu et al. [24] focused on the influence of ambient temperatures on the wear behavior of H13 steel by dry sliding wear tests. The results indicated that the wear mechanism of H13 steel was oxidation wear and the wear rate increased rapidly when the ambient temperature was higher than 600 K and the load was greater than 150 N. In Reference [23], the nitrided layer thickness of H11 steel reaches 143.6 μm at 773 K nitriding temperature. Moreover, the wear rate of nitrided H11 steel reaches $3.2 \times 10^{-5} \text{ mm}^3/\text{mm}$ at 25 N/1500 m friction condition.

According to the above studies, it can be seen that the friction conditions have significant effects on the wear properties of the specimen. Some researchers investigated the effects of the friction conditions on the wear properties of the specimen separately, or simultaneously considered the sliding speed, load and temperature during the traditional plasma nitriding. However, the nitriding temperature on the specimen may be not precise and uniform in traditional plasma nitriding. There is no study about the effects of friction test parameters on the wear properties of specimens treated by auxiliary heating plasma nitriding. This paper proposes an auxiliary heating method to maintain a uniform specimen temperature and precisely control the nitriding temperature during plasma nitriding. Understanding the wear properties of specimens treated by auxiliary heating plasma nitriding under different friction conditions is vital to the mechanical structure design and actual use of the friction parts.

2. Materials and Methods

The specimens with different diffusion layers and different hardnesses are fabricated through changing the plasma nitriding temperature. The surface properties (including surface microstructure, cross sectional morphology, phase and microhardness distribution) of plasma-nitrided specimens are characterized by scanning electron microscope (SEM), X-ray diffractometer, metallographic microscope and microhardness tester. The wear properties (including wear mechanism, friction coefficient, wear rate) of untreated and plasma-nitrided specimens are investigated through friction tests which are conducted on a wear tester with reciprocating sliding wear under dry friction conditions.

2.1. Materials

AISI H11 steel is chosen as the experimental specimen material. The chemical composition of AISI H11 steel is shown in Table 1. The data of chemical composition in Table 1 is from the producers of AISI H11 steel. AISI H11 steel is one of the widely used hot working die steels because of its excellent comprehensive performance, such as toughness, mechanical strength, thermal fatigue behavior, thermal stability and corrosion resistance to liquid aluminum [25]. It is usually used in mold, disk, roll, gear and other products which require high hardness and toughness to bear mechanical wear and thermal shock during working processes. In addition, it is also used in aircraft, rockets and other heat resistant 673–773 K operating temperature structural parts due to its good medium temperature strength. For example, the overrunning spring clutch is a core part in the main driving system of modern helicopters, which supports the transmission of power in one direction [26]. Thereinto, the variable section spring is the key part of the overrunning spring clutch which is usually fabricated by AISI H11 steel. In the transcendental mode, the contact surface between the spring and housing bears huge compressive stress and contact friction [27,28]. Long-term friction will result in the decline of the clutch motivation performance or even failure. Wear failure has become the main cause of clutch failure.

Table 1. The chemical composition of AISI H11 steel.

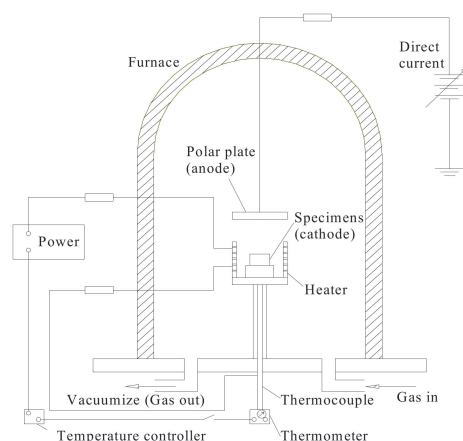
Element	C	Si	Mn	Cr	Mo	V	P	S	Fe
Content (wt %)	0.4	0.85	0.42	4.96	1.18	0.45	0.002	0.014	Balance

2.2. Heat Treatment

The wire electrical discharge machining method is employed to make specimens. The specimens consist of a disc specimen and a cubic specimen. The diameter and thickness of the disc specimen are 50 and 5 mm, respectively. The size of the cubic specimen is 10 mm × 10 mm × 10 mm. The specimens are quenched at 1303 K in a vacuum, and then they are tempered firstly at 853 K for 45 min and secondly at 833 K for 45 min. The average hardness of specimens after heat treatment is 530 HV_{0.2}. Next, the specimen surface is polished by 200#, 400#, 1000# and 2000# metallographic sandpaper in turn. The ultrasonic cleaning method is adopted to wash the specimens for 10 min by anhydrous ethanol. Then, a hair dryer is used to dry the specimens.

2.3. Plasma Nitriding

The plasma nitriding experiment is conducted on the PINI-50 nitriding furnace. Figure 1 represents the working principle diagram of auxiliary heating plasma nitriding. It mainly consists of a direct current power supply, furnace, temperature control system, polar plate, vacuum pump and ammonia pump. The temperature control system is a closed loop control system, which includes a high frequency AC power supply, temperature controller, thermometer, thermocouple and electromagnetic induction heater. The frequency of high frequency AC power supply is 1×10^5 Hz. The principle of the temperature controller is Proportion Integration Differentiation (PID) control. The resolution and measurement range of thermocouple are 0.1 K and 273–873 K, respectively. The shape of the electromagnetic induction heater is cylindrical. The specimen is placed at the center of electromagnetic induction heater. According to the theory of electromagnetic induction heating, the temperature of the specimen surface is basic and uniform. The measurement results of thermocouples also prove this inference. The differences between 8 thermocouples are about 5 K. Namely, the auxiliary heater can effectively maintain a uniform specimen temperature and precisely control the nitriding temperature during plasma nitriding.

**Figure 1.** Working principle diagram of auxiliary heating plasma nitriding.

The procedures of auxiliary heating plasma nitriding are as following: (1) Preparation work: the polar plate and specimen connect anode and cathode, respectively. The air pressure in furnace is reduced to 15 Pa by vacuum pump. The temperature in the furnace can be set by operation system. Then, ammonia gas flows into the furnace whose pressure is maintained at 300 Pa. (2) Formation of a plasma zone: The strength of the electric field between the polar plate and the specimen can reach a

very high level when the direct current power supply works. Then, the ammonia molecule can be ionized to a plasma zone between the anode and the cathode due to the effect of high temperature environment and electric field. The ratio of nitrogen to hydrogen atoms is 1:3. (3) Infiltration of nitrogen atoms: the positive ions of nitrogen bombard the specimen surface at high speed under the strong electric field. The kinetic energy of ions can also be converted to heat energy. Then, nitrogen atoms can adhere on the specimen surface and diffuse into the interior of specimen. (4) Continuously nitriding: ammonia gas pressure, direct current and the temperature in the furnace should be maintained stable for a long time to obtain a certain depth of nitriding layer. In our previous experiment [28], 40Cr steel was chosen as the specimen material, and it was found that plasma nitriding with auxiliary heating could make the hardness distribution more uniform. More specifically, this method could decrease the hardness differences on the whole specimen surface from 81 HV_{0.1} to 21 HV_{0.1}.

According to our experience, the effect of plasma nitriding acting on specimens is difficult to further enhance when the nitriding time is longer than 20 h. In this experiment, the nitriding time is set as 20 h. The nitriding temperature is set at 753, 783 and 813 K.

2.4. Testing Methods

2.4.1. Surface Morphology

The surface microstructure of specimens treated by plasma nitriding is observed by SEM (Tescan, MIRA3, Brno, Czech Republic). The accelerated voltage of electron beam is 20.0 kV. The magnifications are 5000× and 10,000×.

2.4.2. Cross-Sectional Morphology

The preparation for the metallographic specimen of the cross-sectional morphology of the plasma-nitrided specimen is similar to that of a standard metallographic specimen, which mainly includes cutting, inseting, grinding, polishing and etching. The cross-section of the plasma-nitrided specimen is etched by a weak solution of nitric acid and alcohol. The volume content of the nitric acid is 4%. The cross-sectional morphology of the plasma-nitrided specimen is observed through the M-4XC (Lab Testing Technology Co., Ltd., Shanghai, China) metallographic microscope.

2.4.3. Phase Analysis

The phase analysis of the plasma nitrided specimens is investigated by means of X-ray diffraction (Bruker D8 ADVANCE, Karlsruhe, Germany). Cu K α radiation with 2 θ angle 35°–90° and scanning step length 0.02° per step are used.

2.4.4. Microhardness

The microhardness tests of the plasma-nitrided specimens are investigated on HXD-1000TMB micro Vickers (Taiming Optical Instrument Co., Ltd., Shanghai, China). Load is 200 g, loading time is 15 s. The microhardness of three points at the same depth is measured, and their average value is treated as the final value. The distance between two adjacent measured points is 30 μ m.

2.4.5. Friction Test

The friction tests are implemented on the CFT-I multifunctional material surface comprehensive performance tester under dry friction conditions. This tester can precisely measure friction coefficient, wear rate, surface roughness and other surface mechanical properties. The ranges of the load, sliding speed and friction temperature of the friction-wear tester are 10–200 N, 15–300 mm/s and 293–573 K, respectively. The friction forms of the friction-wear tester include rotation, reciprocation and ring block. The hardness of the counter sample is 62 HRC.

The method of the reciprocating sliding wear test under dry friction conditions is adopted in this experiment, and its schematic diagram is shown in Figure 2. Load, sliding speed and friction

temperature are selected as process parameters in this the friction test. In this test experiment, the loads (F) are 50, 100 and 150 N. The sliding speeds (v) are 16.7, 50 and 83.3 mm/s. The friction temperatures (T) are 298, 343 and 373 K. The friction test continues for 30 min without lubrication. The counter probe is a GCr15 steel ball with a diameter of 6 mm. Table 2 shows the experimental conditions of friction tests.

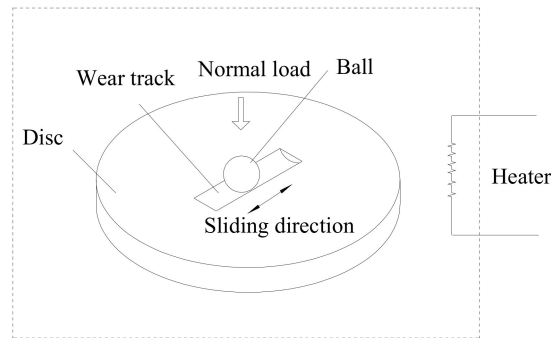


Figure 2. Schematic diagram of ball-on-disc wear test.

Table 2. The experimental conditions of friction tests.

Factor	Specimens	Load (N)	Temperature (K)	Sliding Speed (mm·s ⁻¹)
Normal load	Untreated/Nitrided	50/100/150	298	83.3
Sliding speed	Untreated/ Nitrided	100	298	16.7/50/83.3
Temperature	Untreated/ Nitrided	100	298/343/373	16.7

2.4.6. Wear Trace

The measurement of the wear traces and corresponding wear volume of untreated specimens and plasma-nitrided specimens are studied on the 3-Dimensional Interactive Display (Veeco, Shanghai, China). The wear rate of the specimens is expressed as the wear volume per unit slip distance.

3. Surface Properties

In this section, the surface properties of specimens treated by plasma nitriding are characterized in terms of surface microstructure, cross-sectional morphology, phase analysis and microhardness distribution.

3.1. Surface Morphology

Figure 3 is the SEM image of plasma-nitrided specimen at different magnification rates. The nitriding temperature of this specimen is 783 K. It can be seen that a large number of granular nitrides are generated on the surface of the plasma-nitrided specimens. The diameter of granular nitrides ranges from 0.1 to 1 μm . A portion of the granular nitrides is agglomerated to coralloid granular nitrides. Besides, there are some micro linear grooves on the surface of the plasma-nitrided specimen. This fact may be attributed to the nano scratches from polishing. The granular nitrides preferentially gather on the convex of the nano scratch. Then, the nano scratch can grow into a micro linear groove along with the plasma nitriding time. Compared with Reference [23], this study can obtain the tinier and more uniform granular nitrides on the workpiece surface. The agglomeration of granular nitrides is significantly reduced by auxiliary heating plasma nitriding.

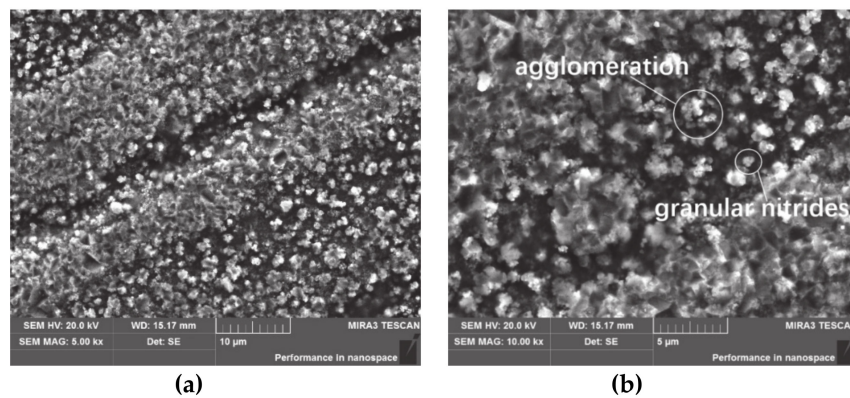


Figure 3. The SEM image of a plasma-nitrided specimen. (a) 5000 $\times$; (b) 10,000 $\times$.

3.2. Cross-Sectional Morphology

Figure 4 shows the cross-sectional morphology of plasma-nitrided specimens. Along the layer depth, the microstructure of the specimen material is a diffusion layer followed by a transition layer and substrate. The thicknesses of the diffusion layers are about 144, 187 and 244 μm when the plasma nitriding temperatures are 753, 783 and 813 K, respectively. Namely, the thickness of the diffusion layer increases with the increase of the plasma nitriding temperature. This fact obeys the basic principle of plasma nitriding. Increasing the nitriding temperature contributes to raising the quantity of nitrogen atoms adhered on the specimen surface, and to enhance the activity of nitrogen atoms. Then, the diffusion speed of nitrogen atoms and the absorption of nitrogen atoms by substrate will be improved. In addition, compared with Reference [23], the proposed auxiliary heating plasma nitriding can obtain thicker nitrided layer than traditional plasma nitriding.

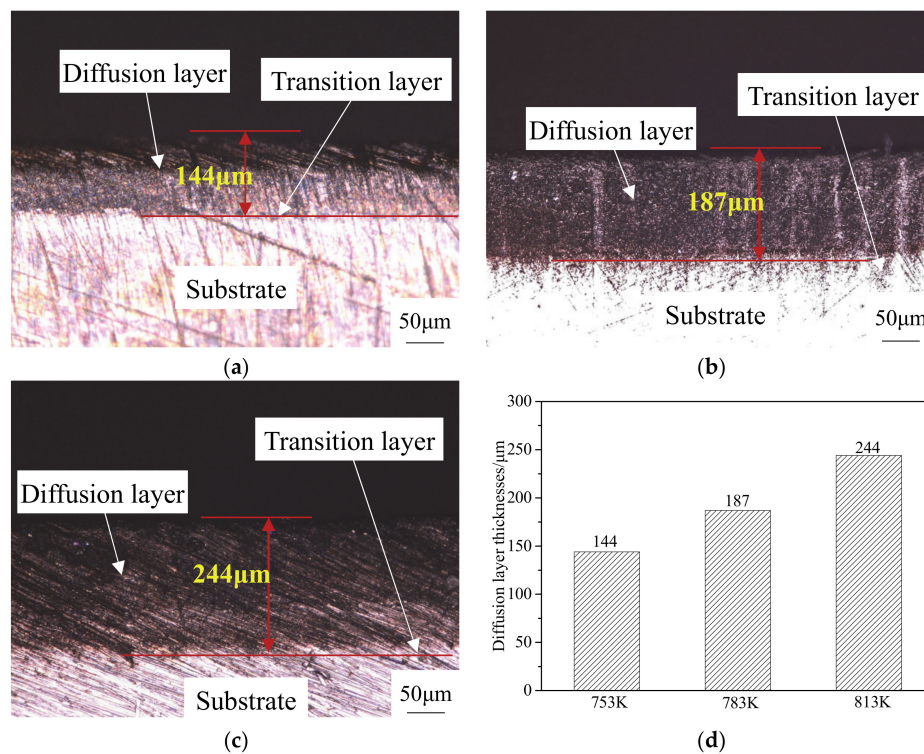


Figure 4. Cross-sectional morphology of plasma-nitrided specimen: (a) 753 K, 500 $\times$; (b) 783 K, 500 $\times$; (c) 813 K, 500 $\times$; (d) diffusion layer thicknesses under different nitriding temperatures.

3.3. Phase Analysis

Figure 5 shows the X-ray diffraction patterns of the plasma-nitrided specimens. The phase composition of the untreated specimen is mainly composed of α -Fe and carbides [29]. M represents the alloying element in AISI H11 steel, such as cementite [30]. The phase compositions of plasma-nitrided specimens mainly consist of γ '-Fe₄N, ϵ -Fe₂₋₃N and CrN. More specifically, the scanning angles of the α -Fe peak, γ '-Fe₄N peak, ϵ -Fe₂₋₃N peak and CrN peak are respectively 44.6°, 41.2°, 43.8° and 36.7°. Besides, the relative content of ϵ -Fe₂₋₃N reduces with the increase of the nitriding temperature. However, the relative content of γ '-Fe₄N rises with the increase of the nitriding temperature. According to Reference [31], the nitrogen potential decreases with the increase of ambient temperature. The higher temperature could give lower nitrogen potential. Then, the more γ '-Fe₄N can be formed in the nitrided layer. In addition, Reference [32] pointed out that increasing plasma nitriding potential could promote the formation of the ϵ -Fe₂₋₃N phase.

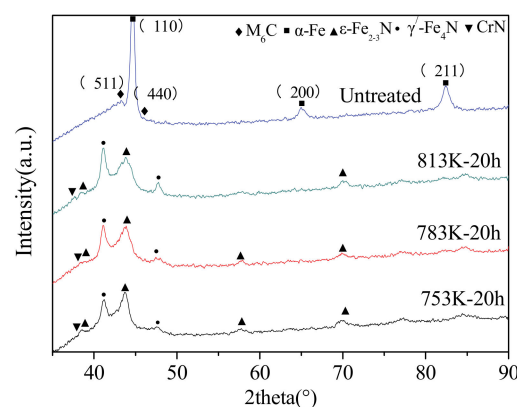


Figure 5. XRD profiles of untreated and plasma-nitrided specimens.

3.4. Microhardness

Figure 6 illustrates the microhardness distribution along the layer depth of the plasma-nitrided specimens. It can be seen that: (1) The microhardness along the layer depth first reduces and then remains stable with the increase of the distance below surface. The steady value is about 530 HV_{0.2} which is close to that of untreated specimen. (2) The maximal microhardness is achieved at the surface of plasma-nitrided specimen. The surface microhardness of the specimens are 1058.8 HV_{0.2}, 1098.7 HV_{0.2} and 1001.6 HV_{0.2} when the plasma nitriding temperatures are 753, 783 and 813 K, respectively. In addition, the microhardness of the specimen material at 0.1 mm depth is higher than 1000 HV_{0.2} when the nitriding temperature is 783 K. Namely, the surface microhardness of the plasma-nitrided specimen first increases and then reduces with the increase of the nitriding temperature. The different nitriding temperatures can obtain different compound structures which are directly related to the surface hardness of the specimen. According to Reference [33], when the nitriding temperature is relatively low, the thickness of the nitriding layer and the diameter of the nitride particles are relatively small. Then, the surface hardness is also relatively low. Along with the increase of the nitriding temperature, the nitriding layer thickness, the nitride's particle diameters and the volume fraction of the nitrides increase. Then, the surface hardness increases. However, when the nitriding temperature is relatively high, the coarse and agglomerated nitride particles can be formed. Then, the surface hardness is decreased. (3) The microhardness at the depth of 0.15 mm is 560.2 ± 19.6 HV_{0.2} when the nitriding temperature is 753 K. The microhardness at the depth of 0.18 mm is 620.2 ± 21.7 HV_{0.2} when the nitriding temperature is 783 K. The microhardness at the depth of 0.18 mm is 651.7 ± 22.8 HV_{0.2} when the nitriding temperature is 813 K. If the measuring errors of the nitriding layer and microhardness distribution are taken into account, the microhardness distribution in Figure 4 shows a good agreement with the cross-sectional morphology observation experiment.

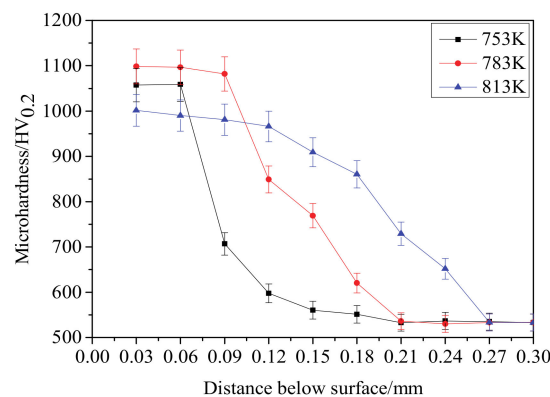


Figure 6. Microhardness distribution of the plasma-nitrided specimen.

From the above characterization analysis of the surface properties of the plasma-nitrided specimens, it can be obtained that the plasma nitriding method can effectively improve the hardness of specimens. The surface microhardness of the plasma-nitrided specimen under 783 K is higher than those under the other two temperatures. Additionally, the microhardness of the specimen material at 0.1 mm depth is higher than 1000 HV_{0.2}. According to previous research [6,17,30,34], the specimen with the higher hardness exhibits the better wear properties in plasma nitriding treatment. Hence, the plasma-nitrided specimen under 783 K is selected as the specimen in the wear properties experiment.

4. Wear Properties

In this section, the wear properties of untreated specimens and plasma-nitrided specimens are analyzed in terms of wear mechanism, friction coefficient and wear rate.

4.1. Wear Mechanism

The wear mechanisms of the untreated specimens and the plasma-nitrided specimens are investigated by the way of SEM observation.

Figure 7 shows the SEM images of the worn surfaces of untreated specimens under different test conditions, loads, temperatures and speeds. As shown in Figure 7a, at 50 N/83.3 mm·s⁻¹/298 K, the wear mechanism of the untreated specimen is typical adhesive wear because there are clear plastic deformations, spalling phenomena and adhesive marks on the worn surface. As shown in Figure 7b at 100 N/83.3 mm·s⁻¹/298 K and Figure 7c at 150 N/83.3 mm·s⁻¹/298 K, when increasing the load from 50 N to 100 N, the wear mechanisms of the untreated specimens are abrasive wear and slight adhesive wear because there are clear scratches, white granular particles, deep pits and slight adhesion marks on the worn surface. As indicated by the comparison results of Figure 7a–c, with the increase of load, the wear mechanisms of the untreated specimens change from adhesive wear to adhesive wear and abrasive wear. In Figure 7a, a deep and narrow groove under low load can be observed. This groove displays low-grade wear in the initial stage under reciprocating sliding wear test. The abrasive wear in the Figure 7c is more serious than that in Figure 7b. According to wear theory [35], adhesive wear often happens before abrasive wear if the wear resistance of the specimen is relatively poor. A mass of worn material due to adhesive wear can easily result in abrasive wear under the relatively greater load. The greater load can lead to the more serious abrasive wear.

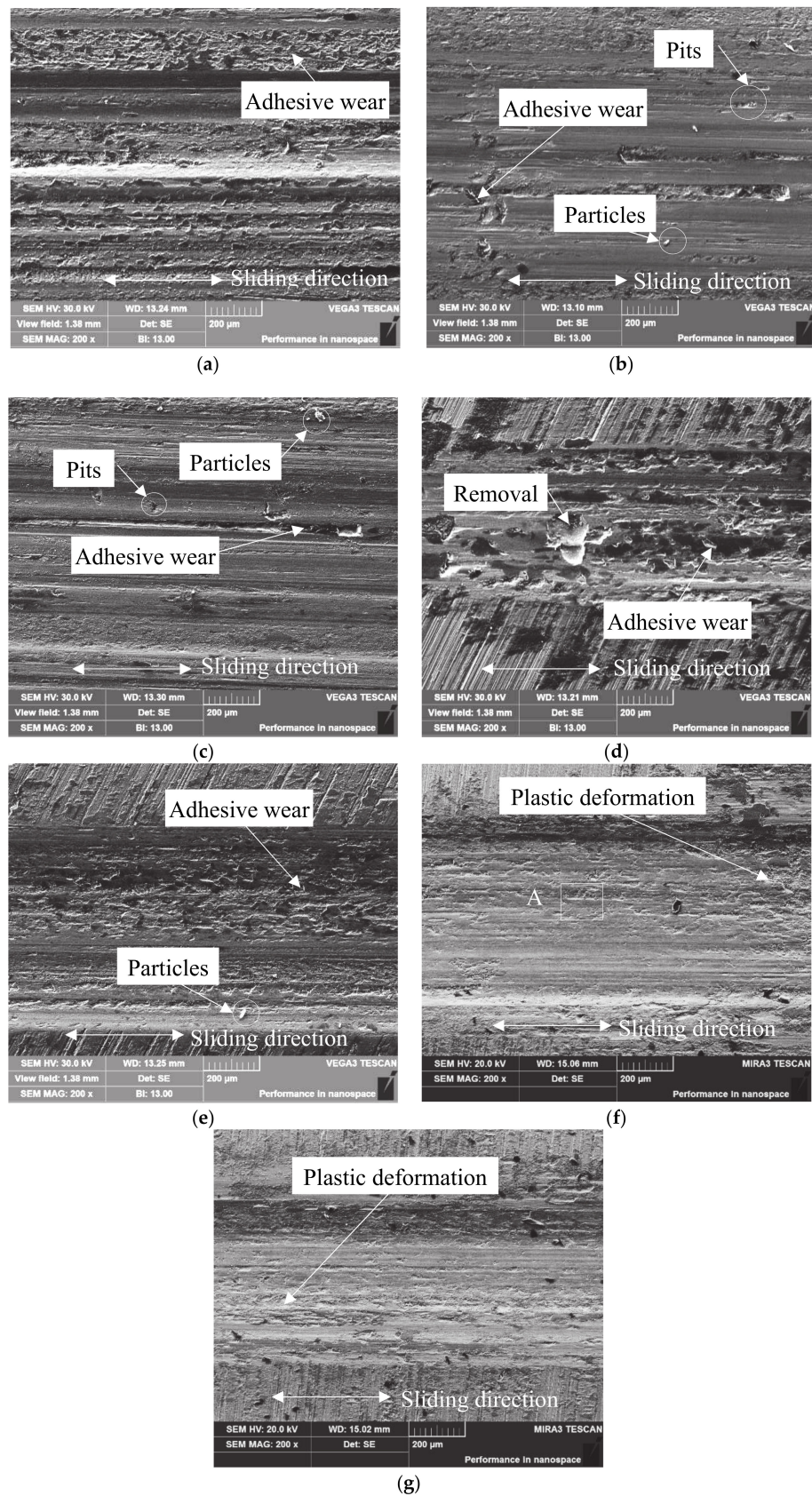


Figure 7. SEM images of the worn surfaces of untreated AISI H11 specimens tested at: (a) 50 N/83.3 mm·s⁻¹/298 K, (b) 100 N/83.3 mm·s⁻¹/298 K, (c) 150 N/83.3 mm·s⁻¹/298 K, (d) 100 N/16.7 mm·s⁻¹/298 K, (e) 100 N/50 mm·s⁻¹/298 K, (f) 100 N/16.7 mm·s⁻¹/343 K, (g) 100 N/16.7 mm·s⁻¹/373 K.

As shown in Figure 7d at 100 N/16.7 mm·s⁻¹/298 K, the wear mechanism of untreated specimens is adhesive wear because there are clear plastic deformations, spalling phenomena and adhesive marks on the worn surface. As shown in Figure 7e at 100 N/50 mm·s⁻¹/298 K, the wear mechanisms of the untreated specimens are adhesive wear and slight abrasive wear, as there are shallow scratches, white granular particles and medium adhesion marks on the worn surface. The comparison results of Figure 7b,d and e indicate that, with the increase of sliding speed, the wear mechanism of the untreated specimen changes from adhesive wear to adhesive wear and abrasive wear. Some fine and shallow scratches are distributed along the sliding direction on the worn surface in Figure 7b,d,e. When the sliding speed is less than 83.3mm/s, some clear adhesion marks appear on the worn surface. When the sliding speed reaches 83.3mm/s, the adhesion mark almost disappears and only local peeling occurs.

The comparison results of Figure 7d,f at 100 N/16.7 mm·s⁻¹/343 K and Figure 7g at 100 N/16.7 mm·s⁻¹/373 K indicate that: with the increase of friction temperature, the phenomenon of plastic deformation is detected. Table 3 shows the EDS analysis of area A in Figure 7f. It can be seen that the number of oxygen atoms can reach up to 33.47%. This fact means that oxidation wear is one kind of the wear mechanism of untreated specimens.

Table 3. EDS analysis of area A in Figure 7f.

Element	Weight %	Atomic %
O	13.45	33.47
Si	0.81	1.14
V	0.69	0.53
Cr	4.61	3.53
Fe	79.16	56.40
Mo	1.28	0.53
Totals	100	-

Figure 8 shows the SEM images of the worn surfaces of plasma-nitrided specimens under different friction conditions. It can be found that the wear mechanisms of the plasma-nitrided specimens are abrasive wear and slight adhesive wear as there are plow grooves, white granular particles and slight adhesive wear traces without shedding phenomenon on the worn surface of the plasma-nitrided specimen. Compared with the worn surface of the untreated specimens, the plastic deformation, spalling phenomena and adhesive wear traces on the worn surface of the plasma-nitrided specimens are reduced. Namely, the plasma nitriding method can significantly improve the wear resistance of AISI H11 steel.

The comparison results of Figure 8a at 50 N/83.3 mm·s⁻¹/298 K, Figure 8b at 100 N/83.3 mm·s⁻¹/298 K and Figure 8c at 150 N/83.3 mm·s⁻¹/298 K indicate that, with the increase of load, the plow grooves and white granular particles become increasingly clearer, while the adhesive wear traces become more and more shallow. There is no visible trace of adhesive marks on the worn surface when the load is 150 N.

The comparison results of Figure 8d at 100 N/16.7 mm·s⁻¹/298 K, Figure 8e at 100 N/50 mm·s⁻¹/298 K and Figure 8b show that, with the increase of sliding speed, the plow grooves and spalling pits become deeper and wider. While, the adhesion marks become less and less noticeable.

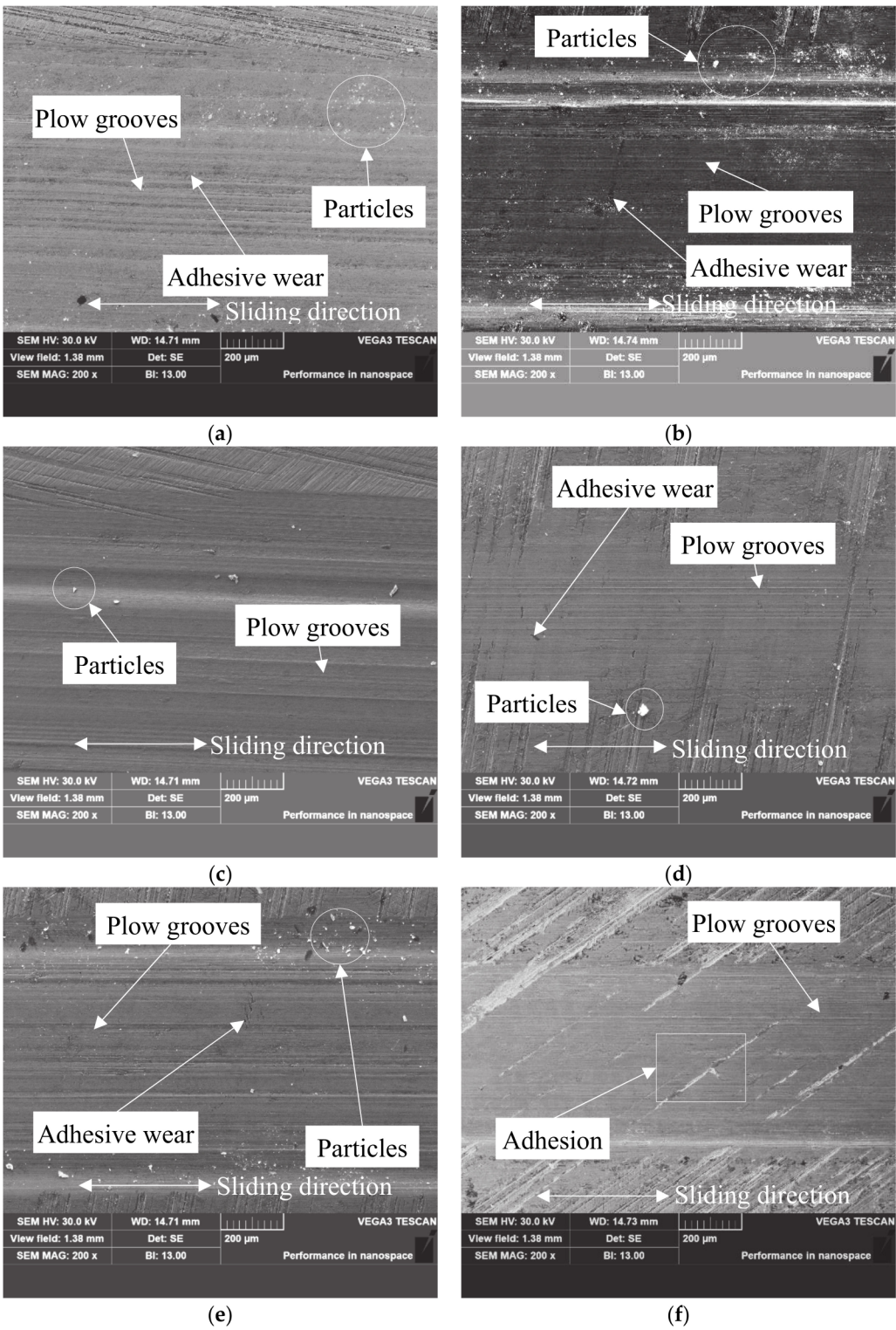
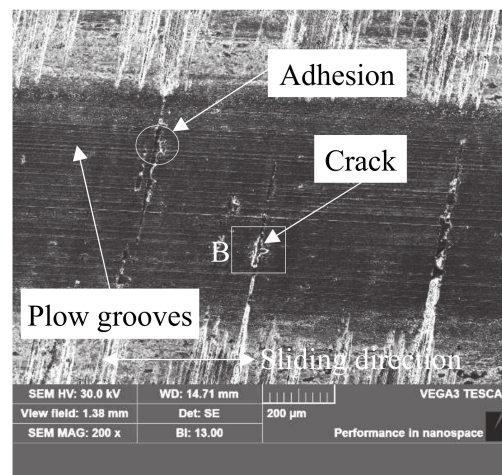


Figure 8. Cont.



(g)

Figure 8. SEM images of the worn surfaces of plasma-nitrided AISI H11 specimens tested at: (a) 50 N/83.3 mm·s⁻¹/298 K, (b) 100 N/83.3 mm·s⁻¹/298 K, (c) 150 N/83.3 mm·s⁻¹/298 K, (d) 100 N/16.7 mm·s⁻¹/298 K, (e) 100 N/50 mm·s⁻¹/298 K, (f) 100 N/16.7 mm·s⁻¹/343 K, (g) 100 N/16.7 mm·s⁻¹/373 K.

The comparison results of Figure 8e,f at 100 N/16.7 mm·s⁻¹/343 K and Figure 8g at 100 N/16.7 mm·s⁻¹/373 K show that, with the increase of friction temperature, the plow grooves and white granular particles become less and less noticeable. In addition, Table 4 lists the EDS analysis of area A in Figure 8g. It can be seen from Table 4 that there is a certain proportion of oxygen element in the area A. However, the proportion of O element in Table 4 is lower than that in Table 3. Namely, the oxidation wear happens on the specimen surface under relatively high friction temperature. Besides, the plasma nitriding method can suppress the oxidation wear.

Table 4. EDS analysis of area B in Figure 8g.

Element	Weight %	Atomic %
O	4.74	14.93
N	4.76	13.02
Si	1.12	1.76
V	0.88	0.76
Cr	4.72	3.99
Fe	82.73	65.07
Mo	1.04	0.48
Totals	100	-

4.2. Friction Coefficient

Figure 9 shows the friction coefficient curves of untreated specimens and plasma-nitrided specimens under different loads. It can be seen from Figure 9 that: (1) The friction coefficients of the untreated specimens are 0.482 ± 0.079 , 0.468 ± 0.084 and 0.444 ± 0.081 when loads are 50, 100 and 150 N, respectively. (2) The friction coefficients of the plasma-nitrided specimens are 0.496 ± 0.063 , 0.474 ± 0.063 and 0.430 ± 0.062 when loads are 50, 100 and 150 N, respectively. Namely, the fluctuations of the friction coefficients of plasma-nitrided specimens are all lower than those of untreated specimens. In addition, the friction coefficients of untreated specimens and plasma-nitrided specimens reduce with the increase of load. As we know, the friction coefficient during adhesive wear is relatively unstable. The friction coefficient during abrasive wear is relatively stable [36]. This fact is consistent with the wear mechanism of untreated specimens and plasma-nitrided specimens.

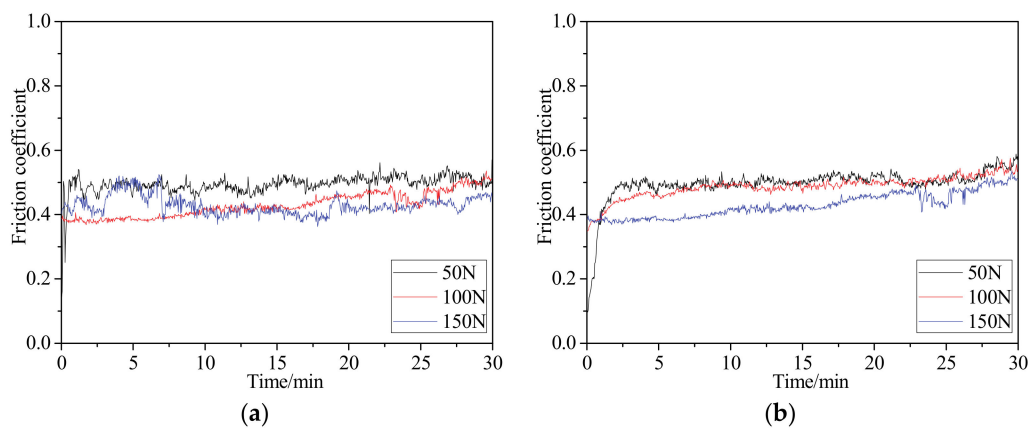


Figure 9. The friction coefficient curves of untreated specimens and plasma-nitrided specimens under different loads: (a) untreated, (b) plasma-nitrided.

Figure 10 shows the friction coefficient curves of untreated specimens and plasma-nitrided specimens under different sliding speeds. It can be found from Figure 10 that: (1) The friction coefficients of the untreated specimens are 0.789 ± 0.144 , 0.641 ± 0.086 and 0.468 ± 0.084 when sliding speeds are 16.7, 50 and 83.3 mm/s, respectively. (2) The friction coefficients of the plasma-nitrided specimens are 0.658 ± 0.084 , 0.579 ± 0.074 and 0.474 ± 0.063 when sliding speeds are 16.7, 50 and 83.3 mm/s, respectively. Namely, the fluctuations of the friction coefficients of plasma-nitrided specimens are all lower than those of untreated specimens. In addition, the friction coefficients of untreated specimens and plasma-nitrided specimens reduce with the increase of sliding speed. This fact is also consistent with the wear mechanism of untreated specimens and plasma-nitrided specimens.

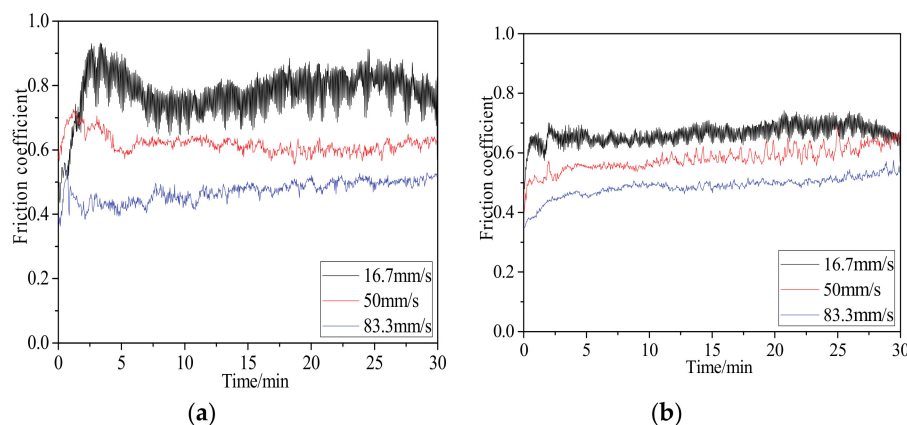


Figure 10. The friction coefficient curves of untreated specimens and plasma-nitrided specimens under different sliding speeds: (a) untreated, (b) plasma-nitrided.

Figure 11 shows the friction coefficient curves of untreated specimens and plasma-nitrided specimens under different friction temperatures. It can be found from Figure 11 that: (1) The friction coefficients of the untreated specimens are 0.789 ± 0.144 , 0.799 ± 0.170 and 0.72 ± 0.100 when friction temperatures are 298, 343 and 373 K, respectively. (2) The friction coefficients of the plasma-nitrided specimens are 0.658 ± 0.084 , 0.623 ± 0.075 and 0.599 ± 0.062 when friction temperatures are 298, 343 and 373 K, respectively. Namely, the fluctuations of the friction coefficients of plasma-nitrided specimens are all lower than those of untreated specimens. In addition, the mean value of the friction coefficients of untreated specimens first increases and then decreases with the increase of friction temperature. This fact is because: the viscosity of untreated specimen increases with the increase of friction temperature, while increasing the friction temperature can also result in softening specimen material [16]. In addition, the friction coefficients of plasma-nitrided specimens reduce with the

increase of friction temperature. This fact is also consistent with the wear mechanism of untreated specimens and plasma-nitrided specimens.

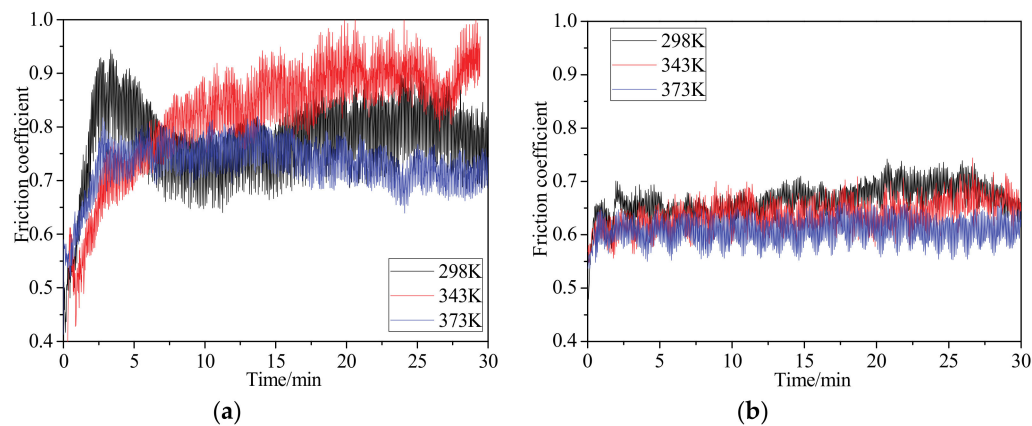


Figure 11. The friction coefficient curves of untreated specimens and plasma-nitrided specimens under different friction temperatures: (a) untreated (b) Plasma-nitrided.

4.3. Wear Rate

Figures 12 and 13 show the three-dimensional white-light interference images of wear traces morphology of untreated specimens and plasma-nitrided specimens. It can be seen that Figures 12 and 13 are consistent with the SEM images in Figures 7, 8 and 14 shows the profile perpendicular to the sliding direction of untreated and plasma treated specimen. The friction parameters are as follows: 50 N/83.3 mm·s⁻¹/298 K.

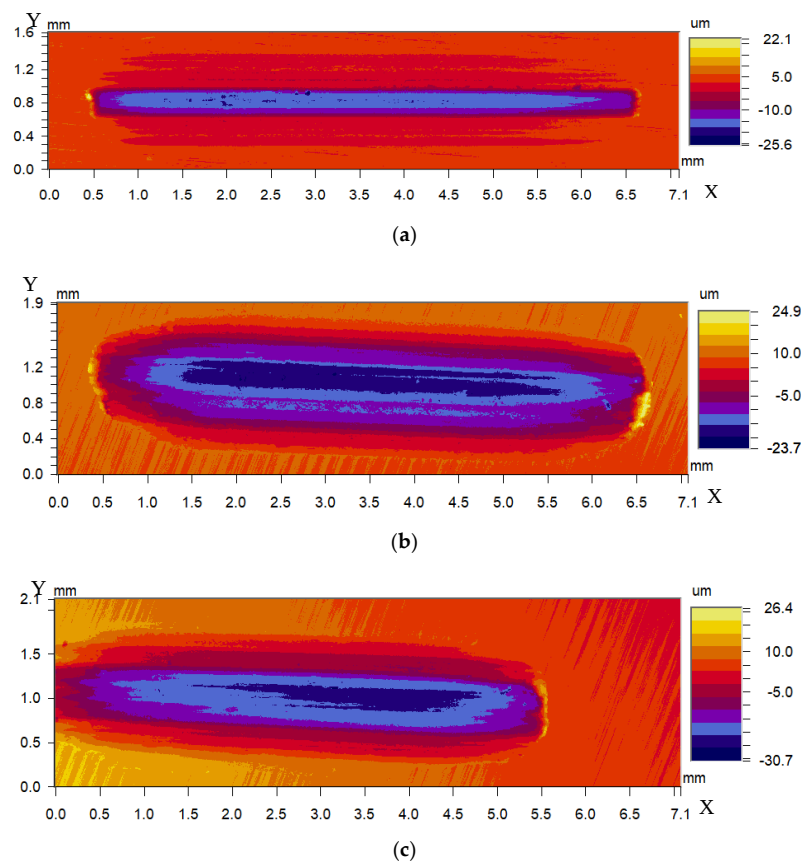


Figure 12. Cont.

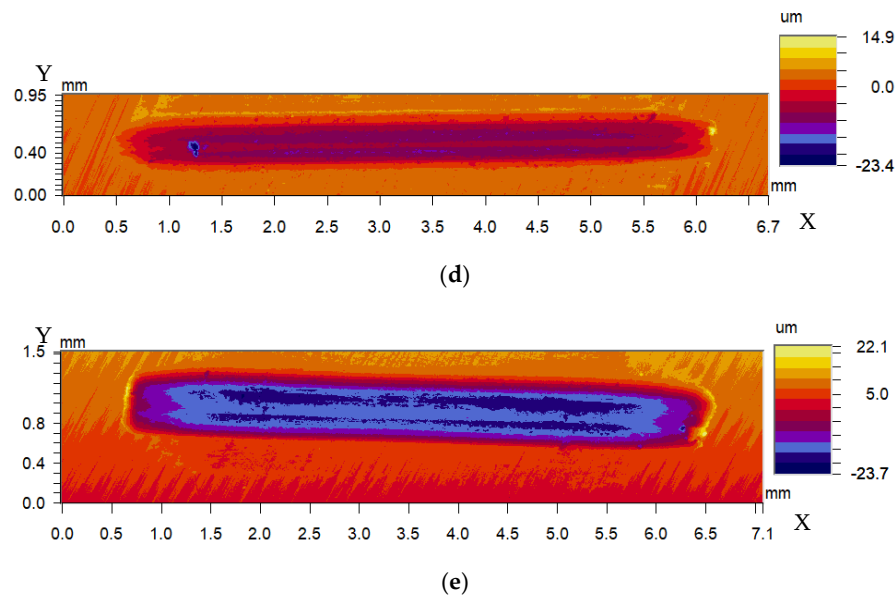


Figure 12. Three-dimensional white-light interference images of wear traces morphology of untreated specimens tested at: (a) 50 N/83.3 mm·s⁻¹/298 K, (b) 100 N/83.3 mm·s⁻¹/298 K, (c) 150 N/83.3 mm·s⁻¹/298 K, (d) 100 N/16.7 mm·s⁻¹/298 K, (e) 100 N/50 mm·s⁻¹/298 K.

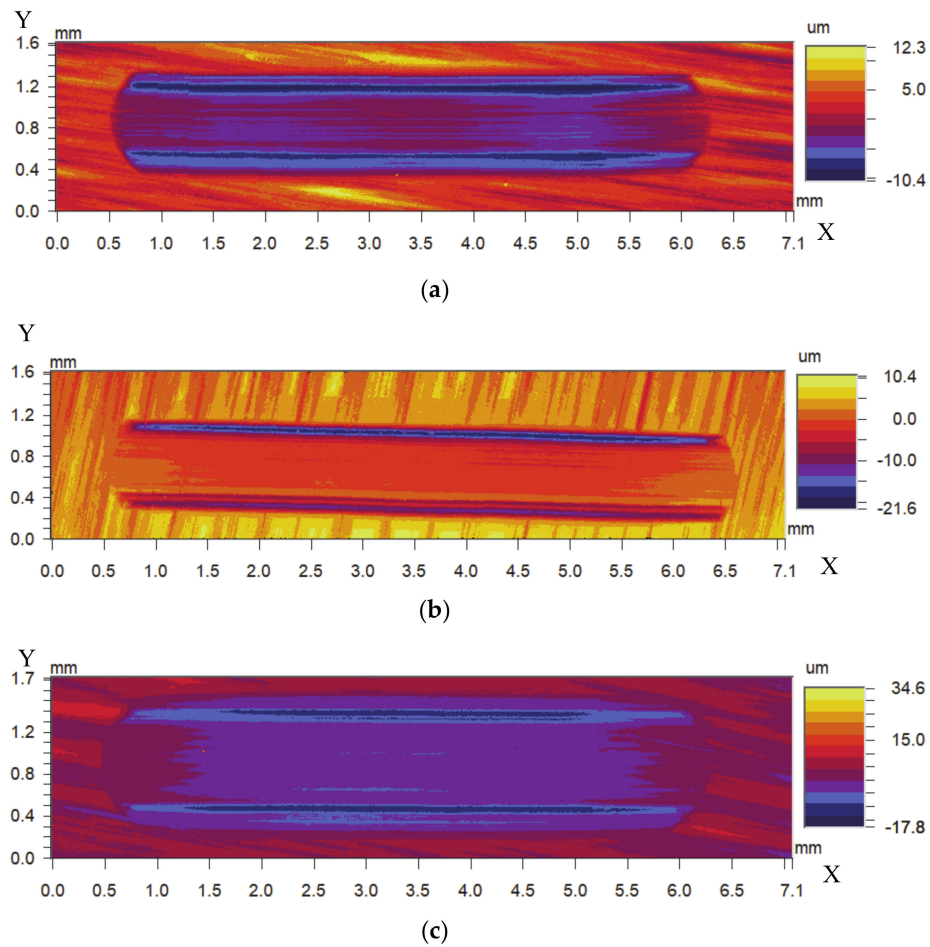


Figure 13. Cont.

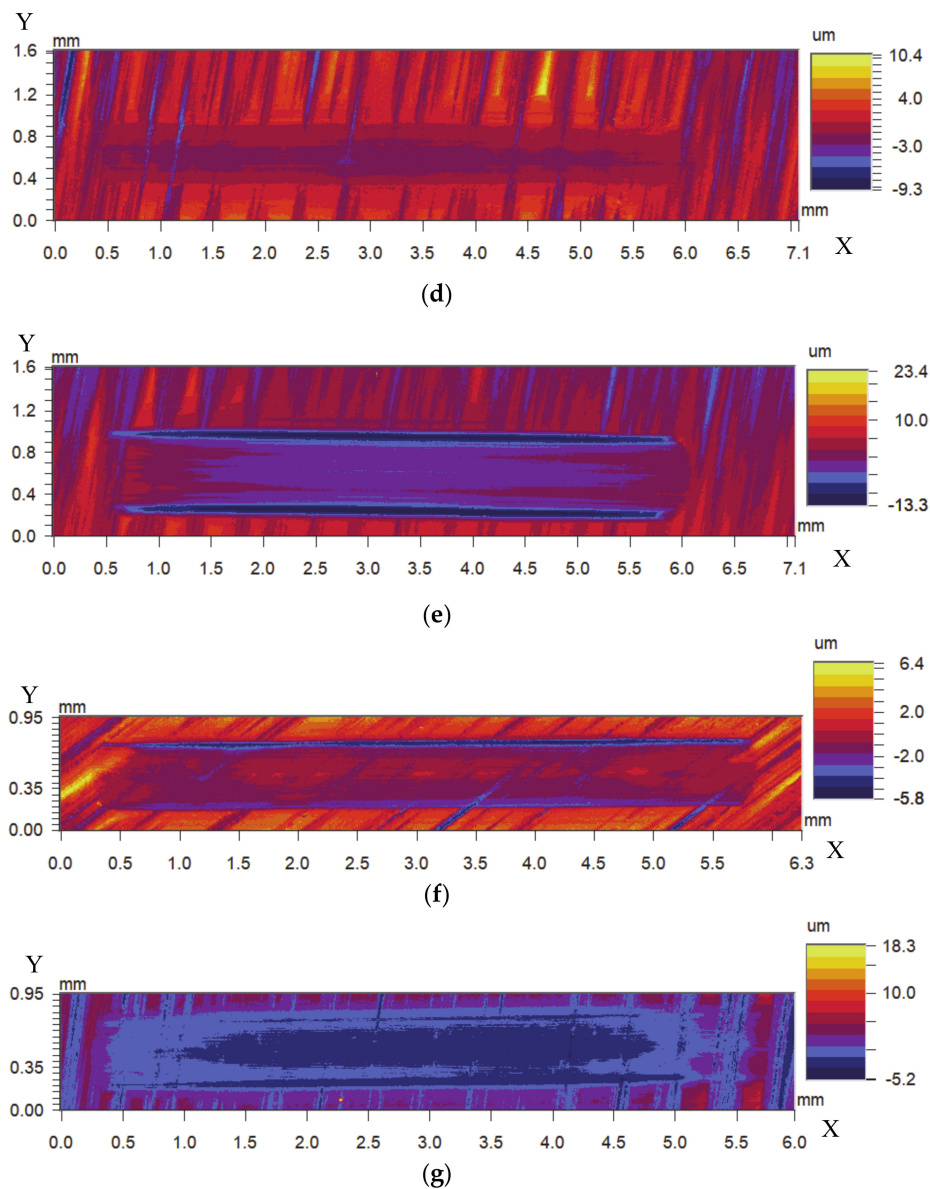


Figure 13. Three-dimensional white-light interference images of wear traces morphology of plasma-nitrided specimens tested at: (a) $50 \text{ N}/83.3 \text{ mm}\cdot\text{s}^{-1}/298 \text{ K}$, (b) $100 \text{ N}/83.3 \text{ mm}\cdot\text{s}^{-1}/298 \text{ K}$, (c) $150 \text{ N}/83.3 \text{ mm}\cdot\text{s}^{-1}/298 \text{ K}$, (d) $100 \text{ N}/16.7 \text{ mm}\cdot\text{s}^{-1}/298 \text{ K}$, (e) $100 \text{ N}/50 \text{ mm}\cdot\text{s}^{-1}/298 \text{ K}$, (f) $100 \text{ N}/16.7 \text{ mm}\cdot\text{s}^{-1}/343 \text{ K}$, (g) $100 \text{ N}/16.7 \text{ mm}\cdot\text{s}^{-1}/373 \text{ K}$.

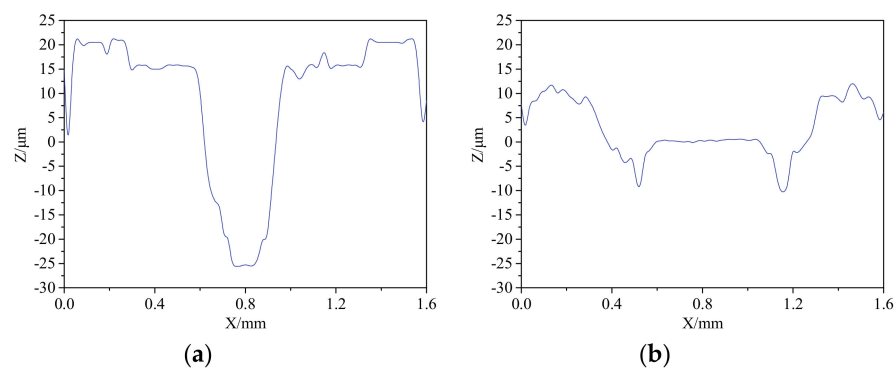


Figure 14. The profile perpendicular to the sliding direction of specimens tested at $50 \text{ N}/83.3 \text{ mm}\cdot\text{s}^{-1}/298 \text{ K}$: (a) untreated, (b) plasma treated.

Figure 15 shows the wear rates of untreated specimens and plasma-nitrided specimens under different loads. The sliding speed in Figure 15 is 83.3 mm/s. It can be observed that the wear rates of untreated specimens range from 1.211×10^{-7} to 2.734×10^{-7} mm³/mm. The wear rates of plasma-nitrided specimens range from 2.85×10^{-6} to 5.64×10^{-6} mm³/mm.

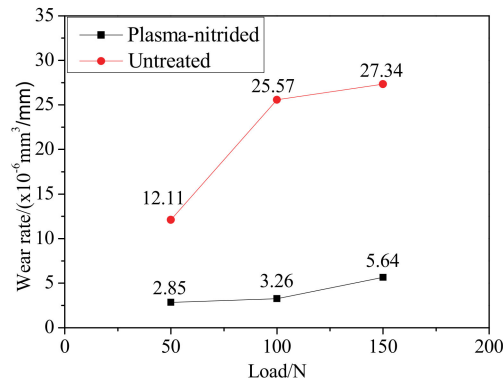


Figure 15. The wear rates of untreated specimens and plasma-nitrided specimens under different loads at the sliding speed of 83.3 mm/s.

The wear rates of untreated specimens first rise relatively quickly and then rise relatively slowly with the increase of load. This fact is because that: as we know, the wear rate during abrasive wear is lower than that during adhesive wear [36]. The main wear mechanism of the untreated specimen is adhesive wear when the load is less than 100 N. The wear mechanism of the untreated specimen is abrasive wear and adhesive wear when the load is 150 N.

In addition, the wear rates of the plasma-nitrided specimens rise with the increase of load. This is attributed to the fact that the increasing load means increasing the plow force. Then, the more specimen material will be removed.

Furthermore, compared with Reference [23], the proposed auxiliary heating plasma nitriding can acquire better wear resistance than traditional plasma nitriding.

Figure 16 shows the wear rates of untreated specimens and plasma-nitrided specimens under different sliding speeds. It can be seen that the wear rates of untreated specimens range from 2.423×10^{-7} mm³/mm to 2.557×10^{-6} mm³/mm. The wear rates of plasma-nitrided specimens range from 3.26×10^{-6} mm³/mm to 7.54×10^{-6} mm³/mm.

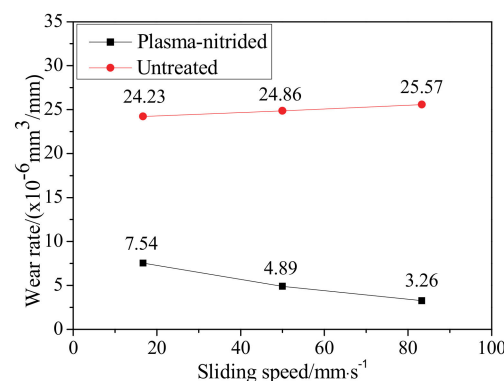


Figure 16. The wear rates of untreated specimens and plasma-nitrided specimens under different sliding speeds at the load of 100 N.

Besides, the wear rates of untreated specimens increase relatively slowly with the increase of sliding speed. In addition, the wear rates of plasma-nitrided specimens drop with the increase of sliding speed. This is because the adhesive wear is dominant when the sliding speed is relatively low, while the abrasive wear is dominant when the sliding speed is relatively high.

Figure 17 shows the wear rates of untreated specimens and plasma-nitrided specimens under different friction temperatures. It can be seen that the wear rates of the untreated specimens range from 2.423×10^{-7} to 4.901×10^{-7} mm³/mm. The wear rates of plasma-nitrided specimens range from 7.18×10^{-6} to 7.54×10^{-6} mm³/mm. From Figures 15–17, it can be concluded that the plasma nitriding method can reduce the wear rate of AISI H11 steel by 68.9~87.3%.

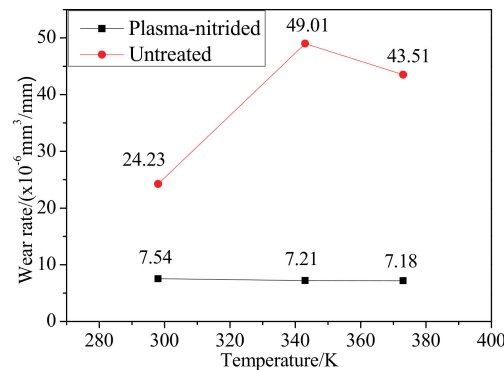


Figure 17. The wear rates of untreated specimens and plasma-nitrided specimens under different friction temperatures at 100 N/16.7 mm·s⁻¹.

Additionally, the wear rates of untreated specimens first increase and then reduce with the increase of friction temperatures. This is attributed to the fact that the hardness and wear resistance will be reduced along with the friction temperature. Hence, the wear rates of untreated specimens under 343 and 373 K are higher than those under 273 K, while the untreated specimen surface is oxidized under high friction temperature. The bonding force between the specimen surface and the oxidation film contributes to suppress the wear of specimen. The oxidation film thickness and the bonding force increase with the increase of friction temperature. That is why the wear rate under 375 K is lower than that under 345 K.

In addition, the wear rates of the plasma-nitrided specimens reduce with the increase of friction temperatures. This is because the nitriding layer is oxidized under the relatively high temperature friction condition. The bonding strength between the oxide film and the matrix increases with the increase of friction temperature. Increasing the friction temperature can also result in softening the specimen material. Then, the white granular particles are easy to embed in the nitriding surface under the relatively high friction temperature. Moreover, the embedded white granular particles can increase the abrasive resistance of the specimen material. Figure 8f also indicates that nitrides are embedded in the metal matrix at high temperatures.

5. Conclusions

Through the above research, the following conclusions can be drawn:

- A large number of granular nitrides are generated on the surface of the plasma-nitrided specimens. The diameter of the granular nitrides ranges from 0.1 to 1 μm. A portion of the granular nitrides is agglomerated to coralloid granular nitrides.
- The thickness of the diffusion layer increases with the increase of the nitriding temperature. More specifically, the thicknesses of the diffusion layer are 144, 187 and 244 μm when the plasma nitriding temperatures are 753, 783 and 813 K, respectively.
- The phase composition of plasma-nitrided specimens mainly consists of γ'-Fe₄N, ε-Fe₂₋₃N and CrN. The relative content of ε-Fe₂₋₃N reduces with the increase of nitriding temperature. However, the relatively content of γ'-Fe₄N rises with the increase of nitriding temperature.
- The surface hardness of the plasma-nitrided specimen is almost twice as high as that of untreated specimen. The surface hardness of the specimen first increases and then reduces with the increase

of nitriding temperature. In addition, the microhardness of the specimen material at 0.1 mm depth is higher than 1000 HV_{0.2} when the nitriding temperature is 783 K.

- The fluctuations of the friction coefficients of plasma-nitrided specimens are all lower than those of untreated specimens. The coefficient of friction reduces with the increase of load as well as with the increase of the speed.
- The plasma nitriding method can significantly reduce the wear rate of AISI H11 steel. The wear rate of the plasma-nitrided specimen was only 12.7%~31.11% of the untreated specimen.
- The wear rates of plasma-nitrided specimens rise with the increase of load, while, the wear rates of plasma-nitrided specimens reduce with the increase of sliding speed and friction temperature.
- Through comparison of the literature results regarding traditional plasma nitriding with our results, the proposed auxiliary heating plasma nitriding can acquire thicker nitrided layers and better wear resistance.

In a word, this study demonstrates that auxiliary heating plasma nitriding can significantly improve the surface hardness and wear resistance of H11 steel. The suitable nitriding temperature for maximal surface hardness is obtained. The influences of friction conditions on the wear resistance are investigated, which provide reference value for the mechanical structure design and actual use of the friction parts, such as mold, disk, roll, gear and variable section spring.

The friction conditions in this study are constant and single. However, the friction conditions in the actual engineering application are complex and ever-changing. In future work, the effectiveness of the suitable nitriding temperature and the influences of friction conditions on the wear resistance should be evaluated in the practical engineering field.

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Conflicts of Interest: The authors declare that they have no conflict of interest.

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