The effect of phase on microstructure and mechanical performance in TiAlN and TiSiN films

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Abstract
To study the effect of phase on the microstructure and mechanical properties of nitride coatings, three films of TiN, TiAlN, and TiSiN were prepared on the surface of high-speed steel using hollow cathode assisted multi-arc ion plating technique. The XRD lines of the three films were analyzed and calculated by linear analysis. The element and phase of the film were observed and analyzed by x-ray Diffraction (XRD), Transmission Electron Microscope (TEM), energy dispersive x-ray analysis (EDS). The microstructure and film thickness of the coating were characterized by scanning electron microscopy (SEM). The surface roughness of the film was observed by Confocal laser scanning microscope (CLSM). The hardness, friction coefficient, and coating/substrate adhesion of the film were tested by the G200 nanometer hardness tester and CETR UNMT-1 surface micro-nano mechanical test system. We discovered two different reinforcement mechanisms. The high microscopic strain value \((1.309 \times 10^{-3})\) in the TiAlN film was related to the formation of Ti₃AlN substitutional solid solution in the film formed a large lattice distortion, however, the coating/substrate adhesion (33.5 N) was lowered. The result of independent nucleation and growth of the Si₃N₄ phase in the TiSiN film refines the structure of the film, alleviating the increase of microscopic strain. At this time, the coating/substrate adhesion reaches the highest value of 40 N and the film surface roughness reaches the minimum value of 0.451 μm. The results also show that the TiSiN coating can obtain good coating/substrate adhesion without pre-plating.

1. Introduction
The TiN binary film prepared by Arc ion plating (AIP) has been widely used in the surface coating of materials such as cutting tools, molds and mechanical parts because of its good mechanical properties [1, 2]. Researchers have also developed ternary or multi-layer films such as TiAIN, TiSiN, and TiCrAIN due to the poor oxidation resistance of TiN films at high temperatures [3–7]. When improving the performance of the multi-layer film, the macrostress or microstrain will be changed due to the increase of its strengthening effect [8, 9], which will affect the coating/substrate adhesion. For the film prepared by the AIP method, the thermal stress in the macrostress [10] has a greater impact. However, in the research work on the ternary films of TiAIN and TiSiN, it is found that the former requires pre-plating TiN on the steel substrate to ensure the bonding force between the TiAIN film and the substrate. However, the difference in thermal expansion coefficient between Si₃N₄ and Fe is much larger than that between Ti₃AlN and Fe, but it does not require pre-coating, and can maintain good coating/substrate adhesion. This phenomenon shows that the macroscopic strain is not the only factor that affects the film-substrate binding force. When the conditions change, the microscopic strain will also have a greater impact on it. The addition of Al and Si elements in the TiN film will produce different phases, which will bring different micro-strain values, different grain sizes and lattice distortions, the effect of the change on the membrane-based binding force is also different.
Table 1. Main process parameters of samples prepared by multi-arc ion plating method.

| Variable                | TiN   | TiAlN  | TiSiN  |
|-------------------------|-------|--------|--------|
| Hollow cathode cleaning current (A) | 120   | 120    | 120    |
| Argon gas pressure (Pa)  | $5 \times 10^{-1}$ | $5 \times 10^{-1}$ | $5 \times 10^{-1}$ |
| Pre-treatment voltage (V) | 240   | 240    | 240    |
| Pre-treatment time (minute) | 30    | 30     | 30     |
| Temperature of substrate (°C) | 300   | 300    | 300    |
| Cathode current (A)      | 80–100| 90–120 | 80–100 |
| Duty ratio               | 80%   | 80%    | 80%    |
| Argon/N2 gas ratio       | 1:30  | 1:40   | 1:50   |
| Working pressure (Pa)    | 1.5   | 1.5    | 1.5    |
| Deposition time (min)    | 60    | 60     | 60     |
| TiN Pre-plating          | no    | yes    | no     |

XRD linear profile analysis (LPA) based on x-ray diffraction technology can be used to not only qualitatively and quantitatively analyze the material phase but also obtain grain structure information such as grain size, lattice parameters, and dislocations [11, 12]. Therefore, researchers have carried out corresponding research work through a variety of methods such as theoretical calculations, experimental detection and pre-plating [13, 14]. In this work, XRD linear profile analysis method [15–18] was used to analyze and calculate the microstrains of the three layers of TiN, TiAlN and TiSiN films prepared by hollow cathode assisted multi-arc ion plating. The calculation results were verified by coating/substrate adhesion detection. At the same time, the cause of micro-strain in the film layer and the strengthening mechanism of TiAlN and TiSiN ternary films are also discussed. In terms of theory and experiment, the results can provide some help for the improvement of the film-based binding force of the TiN ternary film prepared by the physical vapor deposition (PVD) and the microstructure of the coating was studied by means of XRD, SEM and EDS.

2. Experimental procedure and theoretical analysis

2.1. Experimental materials and sample preparation

TiN, TiAlN, TiSiN coating was prepared onto W6Mo5Cr4V2 alloy (HSS) substrate using hollow cathode assisted multi-arc ion plating technique. HSS (15 mm × 15 mm × 5 mm, 63HRC) was chosen as the substrates and they were all separately ultrasonically cleaned in acetone and alcohol for 20 min. The coating equipment was a hollow cathode and multi-arc ion plating composite coating system manufactured (Shenzhen Nissin Vacuum Technology Co., China). Before deposition, the base pressure was pumped less than $8 \times 10^{-3}$ Pa and the test temperature of 300 °C were the preconditions. Then, Ar ion bombardment was employed for 30 min with hollow cathode to remove impurities and activate the surface. The hollow cathode current 120A. The three kinds of films were pre-plated according to the parameters of Table 1, and the targets were pure Ti targets, Ti/Al (50/50), and Ti/Si (82/18) alloy targets, the shortest target-substrate distance is 150 mm. The key of the parameters was to obtain the same coating thickness to reduce the effect of coating thickness on strain.

2.2. Characterization

X-ray diffraction (D-MAXIIA Rigaku) with a wavelength of 0.15406 nm Cu-K$_\alpha$ radiation was used for analyzing the crystallographic phase transformations in the coatings (50 kV, 200 mA, a grazing angle of 1.5° and a scanning speed of 1° min$^{-1}$ in the range of 20° ~ 90°). The surface roughness of the film was observed by LSM700 Confocal laser scanning microscope (CLSM). The cross-section morphology and the thickness of the film was observed by scanning electron microscopy (SEM). The element composition of the coating was determined by a built-in energy dispersive x-ray spectroscopy (EDS), The voltage is 25kV, and the distance between the detector and the sample is about 1.15mm system. electron diffraction analysis was conducted by transmission electron microscopy (TEM). Oliver and Pharr method [19] was used to extract values of hardness and elastic modulus from the G200 nanometer hardness tester (Agilent Technologies, diamond Berkovich indenter. Depth Limit:100nm, five indentations were investigated on each thin film). Besides, the coefficient of friction and the coating/substrate adhesion was obtained with by CETR UNMT-1 surface micro-nano mechanical test system. The grinding material was a GCr15 ball with a diameter of 4mm. The reciprocating sliding friction was...
maintained at a speed of 5 mm s\(^{-1}\) under a load of 1N. The length of the scratch track was 10 mm, the room temperature was 20 °C, and the relative humidity is 50%. The scratch adhesion test was performed with a triangle diamond indenter (60° cone) by continuously increasing the load with a normal force from 1 to 70 N, when the diamond indenter scratches the film and contacts the substrate. The acoustic signal curve and friction coefficient curve will change suddenly. At this time, the corresponding load can be characterized as the coating/substrate adhesion. COF is the coefficient of friction and AE is the acoustic signal.

2.3. Strain measurement and x-ray diffraction linear analysis
Generally, the material would undergo lattice distortion and grain size change during the preparation process; which would broaden the physical line shape of the XRD diffraction line. Since the contribution of these two factors to the diffraction peak broadening is independent, the diffraction line broadening is a superposition of the two broadening effects, as shown in figure 1; the coordinate origin \(O\) is located at \(20_{hkl}\), 1 represents the grain size linear \(f_D(x)\), 2 represents \(f_D(x)\) divided into several narrow bars, 3 represents expanding the narrow bars according to \(f_D(x)\), and 4 represents the final synthetic physical measurement line \(f(x)\). Then, the physical broadening profile \(f(t)\) can be represented by the convolution of the grain size linearity \(f_D(x)\) and the microscopic distortion line shape \(f(x)\), that is: \(f(t) = \int_{-\infty}^{\infty} f_D(x)f_D(t-x)dx = f_D(x) \cdot f_D(x)\), where \(\cdot\) is the convolution symbol. In order to obtain physical and instrumental parameters, XRD diffraction curves must be separated. Therefore, the experimental XRD line shapes can be fitted by convolution of the instrument and sample functions.

Wang method [20] has the following advantages: (1) For the comprehensive analysis of various defect configurations, this method is suitable for Powder and anisotropic bulk polycrystalline industrial materials. (2) It has made a set of standard curves for the three common structures of fcc, bcc and hcp, which can easily measure the grain size and microstrain.

Experimental observations show that the broadened line shape caused by grain refinement is close to the Cauchy line shape while the broadened line shape produced by microstrain is closer to the Gaussian line shape. Then, \(f(x)\) is obtained by the Fourier deconvolution method given by Stokes. Generally, the trigonometric form transformation is used in the calculation process, and the entire interval is averaged by 60 parts for calculating the Fourier transform coefficients. Therefore, the physical broadening profile \(f(x)\) can be expressed as [18]:

\[
f(x) = \sum (A_n \cos 2\pi nx/60 + B_n \sin 2\pi nx/60)
\]

(1)

\(A_n\) and \(B_n\) are the cosine and sine parameters, respectively. \(A_n = Nn/N_3(\cos 2\pi mZ_n), B_n = Nn/N_3(\sin 2\pi mZ_n)\). Among them, \(N_3\) is the average unit cell number, \(n\) is the number of unit cells in the interfering unit cell distance, \(N_n\) is the logarithm of \(n\) unit cells, \(Z_n\) is the microscopic distortion reference quantity, and \(m\) is the reflection order. After \(A_n\) and \(B_n\) are normalized, \(A_0 = B_0 = 1\). It should be noted that \(A_n = A(L), L = 1;\) where \(L\) is the distance or length of coherent unit cells, \(t\) is the harmonic number, \(t = 0, 1, 2, 3\ldots\) is the reflection order, following the Warren-Averbach method:

\[
B(L) = B^p(L)B^l(L)
\]

(2)

\[
A(L) = A^p(L)A^l(L)
\]

(3)

\(A(L)\) is the Fourier transform function of the diffraction curve; \(A^p(L)\) and \(A^l(L)\) are the function of strain broadening and grain size broadening.
Among them, $a$ is the hook coefficient, $D$ is the coherence domain size, $\beta_c$ and $\beta_g$ are the integer widths of the Cauchy and Gaussian functions, $N$ is the average total unit cell number in the same ingot, and $D_{off}$ is the unit cell length dimension ($Nd = D_{off}$). According to a series of values of a single profile $A^L(L)$, $a$, $D$, $\beta_c$, $\beta_g$, and microscopic strain $\langle \varepsilon_L^2 \rangle$ can be calculated using the least-squares fitting program and the corresponding microstrain $\langle \varepsilon_L^2 \rangle$ can be obtained using LASF software.

3. Results and discussions

3.1. Effect of phase composition of the film on its microstrain
The elemental composition of the coating is shown in figure 2. In addition to the elements of the film layer, the elements of the high-speed steel substrate appear due to the thin coating. Considering the limitations of EDS quantitative analysis of the element N, only the main elements of the film layer are listed, the atomic ratio of Ti / Al is 3.95, and the atomic ratio of Ti / Si is 3.3.

Figure 3 shows grazing incidence XRD diffraction line of three films of TiN, TiAlN, and TiSiN. Analysis with Jade software and EDS spectrum analysis, the main phase compositions of the films are TiN, Ti$_3$AlN and TiN, TiN and Si$_3$N$_4$. 

$$A^L(L) = a - L/D_{off}$$

$$A^L(L) = \exp \left[ -2/3cL - \pi (\beta_g)^2L^2 \right]$$

$$\ln A^L(L) = -2\pi^2m^2n^2\langle \varepsilon_L^2 \rangle$$

Figure 2. EDS spectra of two coatings, (a) TiAlN, (b) TiSiN.

Figure 3. XRD diffraction lines of three different sample films that are prepared: (a) TiN peak positions, (b) TiN film, (c) TiAlN film, (d) TiSiN film.
In order to improve the accuracy of the calculation results, the x-ray tube voltage and current are increased, and the scanning speed is reduced when XRD detection is performed on the three film layers. In addition, the average of four strong diffraction peaks (111, 200, 220, 311) of TiN in the three film layers was calculated [21]. The results calculated by the above method are shown in table 2.

It can be seen from the calculation results in table 2 that the order of the microstrains in the three film samples prepared is TiAlN > TiSiN > TiN. The micro-stress state in the film can be represented by this relationship and the phase composition in the film would influence this. According to the XRD diffraction line analysis of the sample of figure 3, the main constituent phases in the TiAlN film are Ti3AlN and TiN. According to thermodynamic data, since Ti radius (0.145 nm) and Al atom radius (0.143nm) are close, relative difference of 2% is much smaller than the solid solubility limit atoms relative difference of 15% [22]. TiN (−308.3 KJ/mol) and Ti3AlN (−314.42 KJ mol⁻¹) are much smaller than the Gibbs generation free energy values of AlN (−287.0 KJ mol⁻¹) and TiAl3 (−170.45 KJ mol⁻¹) [23]. Therefore, two phases of TiN and Ti3AlN are preferentially formed in the ternary system, and Ti3AlN is a substitutional solid solution in which Al is dissolved in the TiN lattice to occupy the position or vacancy of the Ti atom node. The difference in atomic radius causes the formation of such solid solutions with large distortion. All of these will lead to the increase of strain in the film. In the TiSiN film, the Si atom radius (0.118 nm) is significantly different from the Ti atom radius (0.145 nm), the relative difference is 18% greater than 15%. The greater lattice distortion can be caused by forming a substitutional or interstitial solid solution. Thermodynamically, it is in a very unstable state and is difficult to achieve. Fuentes, Gonzalo G et al [24] have studied that only when the Si content is less than 3at%, Si will form a solid solution in TiN, at this time, the grains of TiN will not be refined. There are also studies suggesting that amorphous Si₃N₄ will be formed in this case [25–27]. In this work, through XRD and TEM diffraction analysis techniques (figures 3 and 4), it was determined that the TiSiN film layer generated two separate phases of TiN and Si₃N₄. Selected area electron diffraction spot, shown in figure 4(b), analysis and calculations confirmed that there are two phases of TiN and Si₃N₄ in the TiSiN coating, which are consistent with the results of XRD diffraction analysis. Although the formation of the Si₃N₄ phase does not lead to lattice distortion caused by solid solution strengthening, the ‘free growth’ of the TiN single phase is hindered, so strain is created in the film, on the other hand, increasing the number of grain boundaries by grain refinement also can relieve strain. Therefore, the value of the strain of the TiSiN film is between the TiAIN and the TiN. The coating/substrate adhesion of the above three film samples was tested in the experiment, and the results are shown in figure 5 and table 3. The order of the numerical values is shown as follows: TiSiN film (40N) > TiN film (39.5N) > TiAlN film (33.5N). For this result, the macrostress cannot be explained clearly. The difference in thermal expansion coefficient between the high-speed steel substrate and Si3N4 is the largest (Fe: 12 × 10⁻⁶ °C⁻¹, SiN₄: 3 × 10⁻⁶ °C⁻¹, TiN: 9.35 × 10⁻⁶ °C⁻¹, Ti₃AlN: 8.8 × 10⁻⁶ °C⁻¹), the coating/substrate adhesion between TiSiN coating and substrate should be the worst among the three coatings, but the actual results are opposite. For this

![Figure 4](image_url)
Figure 5. Three different coating/substrate adhesion strength test curves prepared. (a) TiAlN, (b) TiSiN, (c) TiN.

Table 3. Partial mass and performance test values for three different layers.

| Films | Roughness (μm) | Adhesive strength (N) | Hardness (GPa) | Elastic modulus (GPa) | H/E | Coefficient of friction |
|-------|---------------|-----------------------|----------------|-----------------------|-----|------------------------|
| TiSiN | 0.451         | 40.0                  | 33.9           | 347                   | 0.097 | 0.76                  |
| TiAlN | 0.888         | 33.5                  | 44.8           | 385.6                 | 0.116 | 0.73                  |
| TiN   | 0.941         | 39.5                  | 21.0           | 257.1                 | 0.082 | 0.817                 |
phenomenon, the microstrain calculation results can provide some supplements, with the increase of microstrain in the film, the coating/substrate adhesion will also decrease. On the contrary, it can improve and enhance the membrane-based binding force.

3.2. Mechanical and tribological properties

In the prepared TiAlN and TiSiN films, in addition to the different strain characteristics of the constituent phase, other qualities such as microstructure refinement and surface roughness of the film are also affected by the different properties of the phase, which in turn affects the mechanical properties of the film. The image of the film cross section as shown in figure 6. It can be seen that the growth of TiAlN (figure 6(b)) and TiN films (figure 6(a)) in the form of coarse 'columnar crystals' is affected by the rapid heat dissipation in the direction of substrate. The TiSiN film is also formed into a 'columnar crystals' according to the PVD film formation process, but compared with the former two, it has obvious refinement. The difference sizes of the two microstructures is mainly caused by the different phases that are produced when the film is formed. In addition to TiN, the TiAlN

Figure 6. Cross-sectional SEM micrographs of (a) TiN, (b) TiAlN, (c) TiSiN.
film has a new phase formation of Ti₃AlN. However, it is formed by dissolving Al into TiN to form a substitutional solid solution instead of nucleating and growing alone. On the contrary, in the process of TiSiN film formation, the nucleation and growth of Si₃N₄ are slower than TiN because Si atoms are more difficult to ionize than Ti atoms. When TiN is nucleating and growing, Si₃N₄ phase grows at the interface of TiN, which prevents TiN phase from continuing to grow. The continuation of the film formation process depends on tin nucleation and growth. The structure of the film was refined by this phenomenon. The refinement of the film structure also changes the surface roughness of the film. Figure 7 shows the surface roughness of three different film samples. The specific values are shown in table 3. It can be found from the data in table 3 and the morphology of the pictures in figures 5 and 6 that the surface of the film becomes flat as the surface roughness value decreases. The refinement of the film structure has a significant influence on the TiSiN film, Si₃N₄ can form the refining film structure, and at the same time, it can reduce the surface roughness of the film. This is equivalent to increasing the wear area in terms of the friction pair on the surface of the film, and reducing the load per unit area; this is beneficial for improving the wear resistance of the film. A similar effect can be obtained by reducing the strain in the film when the other conditions are similar.

Figure 8 shows the load-displacement curve for each coated specimen. The shape of the curve differs from one coat to the next, and these variations usually reflect different mechanical properties. Each curve contains two parts for loading and unloading. For all samples, the curve shows an initial increase in displacement with increasing applied load. The difference between the hardness of the samples is obtained from the difference in the maximum indentation depth. Figure 9 and table 3 shows the elastic modulus and the hardness of the coatings. In the TiAlN film, the increase in lattice distortion is caused by the solid solution of Al atoms, intensifying the hindrance to dislocation movement. Therefore, both the film strain and the dislocation density are improved, and the hardness value is higher than that of the TiSiN film and the TiN film. However, although the TiSiN film does not have the solid solution strengthening as the former, the fine grain strengthening effect by the formation of the Si₃N₄ phase in the film also leads to an increase in the dislocation density, and the film also has higher hardness value compared to the TiN film. It is worth noting that from the test data of the three films prepared, the TiAlN film has the highest hardness value of 44.8 GPa and the TiSiN film has the hardness value of 33.9 GPa, both of which are higher than the hardness value of TiN film (21 GPa), the results are basically consistent with similar studies [28, 29]. Comparing the hardness values of the two ternary films, the TiAlN film is about 32.1% higher than the TiSiN film, but the friction coefficients of the two are very close and only about 4% difference and both are smaller than the friction coefficient of TiN coating and substrate (0.817 and 0.86, figure 10). It shows that the friction coefficient of the film layer is not only related to the hardness value of the
Figure 8. Load-displacement curve of TiN, TiSiN and TiAlN obtained by nanoindentation test.

Figure 9. Hardness and modulus of elasticity of different layers.

Figure 10. Friction coefficient of different layers (a) HSS, (b) TiN, (c) TiAlN, (d) TiSiN.
coating, but also related to the surface roughness and micro-strain value of the TiSiN film layer. Grain refinement means that the increase of grain boundaries can increase the resistance to dislocation and increase the hardness. At the same time, compared with the inside of the grain, the atomic binding force between the grain boundaries is smaller, and the grains are easy to rotate or deform when they are stressed, which will release and relieve the micro strain. In addition, the self-lubricating properties of the Si$_3$N$_4$ phase itself also play a role. Moreover, the measured value of the friction coefficient of the film is also in good agreement with the hardness and Young’s modulus ratio (H/E) [30–32], providing a valuable reference data for the wear resistance of the film.

4. Conclusions

Based on the high temperature oxidation resistance of Al and Si elements, in order to improve the performance of TiN films, TiAlN and TiSiN films were deposited on the surface of high speed steel by the hollow cathode assisted multi arc ion plating method. The strain size in the film was calculated by XRD linear analysis method, the quality and main mechanical properties of the film were characterized. Through comprehensive analysis, the main conclusions are as follows.

(1) In the TiAlN coating, a substitutional solid solution Ti$_3$AlN based on TiN was formed. Compared with TiN film, it has high microstrain due to its solution strengthening and relatively coarse columnar crystal structure. The hardness of the film reaches 44.8 GPa, and the distortion also affects the coating/substrate adhesion, with the detection value of 33.5 N.

(2) Different from the above, there is no TiSiN ternary phase in the prepared TiSiN system film, and the film is mainly composed of TiN and Si$_3$N$_4$ phases. The formation of Si$_3$N$_4$ changes the columnar crystal growth mode of TiN preferred orientation and refines the film structure. The calculated value of micro strain is smaller than that of TiAlN coating. The detection value of coating/substrate adhesion was 40 N. The hardness of 33.9 GPa is higher than 21 GPa of TiN.

(3) Based on the analysis of the influence of the macro stress caused by the difference of thermal expansion coefficient between TiAlN and TiSiN on the coating/substrate adhesion, the effect of micro strain in TiSiN coatings is more important. Under the experimental conditions, even if the TiSiN system film is not pre plated, it still has a good coating/substrate adhesion.

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