The varying temperature tribological properties of TiAlN-Ag coatings deposited by magnetron sputtering

Yan Zhao¹, Shengshuang Hu², Changjie Feng¹*, En Chen¹

¹School of Materials Science and Engineering, Nanchang Hangkong University, Nanchang, 330063, China
²AVIC Xi'an Aircraft Industry (Group)Company LTD, Xi'an, 710089, China
*Email: chjfengniat@126.com

Abstract: TiAlN-Ag coatings were prepared on the AISI304 stainless steel by magnetron sputtering. The surface and cross-section morphology, microstructure, hardness and tribological properties of the coatings were investigated by scanning electron microscope(SEM), X-ray diffraction(XRD), micro-hardness tester and tribometer. The thickness of the TiAlN-Ag coating was about 10.2 μm, which consisted of face centered cubic TiAlN phases. Its hardness was 1044.8 Hv0.05. The tribological properties of the coatings were investigated by ball-on-disc tribometer against Al₂O₃ balls at RT-600 °C and 600 °C-300 °C. When at RT-600 °C, the coatings were worn badly, which resulted from that the adsorbed water film was destroyed due to the lower relative humidity. The wear mechanisms were mainly adhesive wear and fatigue fracture. When at 600 °C-300 °C, Ag diffused to the surface of the coating and acted as lubricant.

1. Introduction
TiAlN coatings possessed good oxidation resistance above 800 °C, attributed to the protective Al₂O₃ layer generated on the surface at high temperature[1,2]. Because of that, TiAlN coatings were widely used in the field of mechanical processing replaced TiN coatings from 1950s. Al atoms replaced Ti of the TiN crystal lattice, which increased the lattice distortion and internal residual stress, decreased adhesion strength[3]. The friction coefficient of TiAIN coatings was higher.

Noble metals (such as Ag, Au, etc.) possess lower shear strength and higher stability in wide temperature range, which had been added to carbide[4] and nitride[5,6] superhard coatings. Where hard coating phases provides hardness and soft metals provide lubrication.

At present, most laboratory studies on tribological properties of coatings were conducted at some constant temperatures. Actually, the temperature of coatings covering machine devices which were running would change over time[7]. In this paper, TiAlN-Ag composite coatings were deposited by magnetron sputtering, and its varying temperature tribological properties would be investigated by ball-on-disc tribometer.

2. Experimental Details

2.1 Coatings deposition
TiAlN-Ag coatings were deposited on the 20mm × 30mm × 2mm AISI304 stainless steel sheets, by reactive mangnetron sputtering using Ti₄₅Al₅₀Ag₅ target (99.99% purity). During all the sputtering
process, the substrate temperature, the holder rotational speed, the working pressure and the target power were kept constant at 250 °C, 13 rpm, 0.8 Pa(Ar:N2=1:2) and 550 W, respectively. The TiAlN-Ag coatings deposition time was 300 min. The samples were taken out from the chamber when its temperature decreased to the room temperature after sputtering.

2.2 Coatings characterization
The chemical composition of the coatings were determined by energy disperse spectroscopy[8,9]. The surface and cross-section micromorphology of the TiAlN-Ag coatings were observed by scanning electron microscopy (S-3400N).

The phase formation in the film was investigated by X-ray diffraction(XRD), the XRD patterns were recorded using PHILIPS XPERT-PRO-MRD-A25 X-ray diffractometer using a Cu Kα radiation(λ=0.154 nm).

The hardness of the coatings were characterized by microhardness tester.

2.3 Tribological tests
The tribological properties of TiAlN-Ag coatings were investigated by a ball-on-disc rotating tribometer (HT-1000) at RT-600 °C and 600 °C-300 °C. In this work, a Al2O3 ball (diameter 5 μm) was used as the counterpart. The tests were performed with a sliding speed of 196 rpm under a load of 2.2 N. The rotational radius and sliding time were 3 mm and 20 min, respectively. The wear rate was calculated using the equation: K=V/SF, where S is the sliding distance, V is the wear volume, F is the load 2.2 N. The morphology and the chemical composition of the wear tracks were analyzed by SEM and EDS.

3. Results and Discussions
3.1. Morphology of TiAlN-Ag Coatings
The surface micromorphology was shown as figure 1(a), some particles with diameter 0.5-6 μm were distributed on the coating surface, which resulted from the nucleation of the stable Ag atoms[5]. From figure 1(b), the uniform and denser typical column grains were clearly seen. The thickness of the coatings was about 10.2μm. The EDS composition (at.%) of the coating was Ti 17.61, Al 27.97, Ag 1.21, N 49.39, O 3.82.
3.2. Crystal structure and phase identification

Figure 2 shows the XRD diffraction spectra of the TiAlN-Ag coatings. The coating mainly consisted of Face-centered cubic TiAlN phases with preferred orientation of (111). The peaks at 38.65° and 79.3° mainly consist with oxide, which resulted from the contamination of O released from the chamber during deposition.
3.3. Tribological properties

Because the coatings were worn out at 12 min, the samples, which were worn 10 min, were employed to investigate the wear mechanism of TiAlN-Ag coatings. As shown in figure 3, the friction coefficient increased with the time. The adhesion wear and abrasive wear were characterized by the wear track morphology in the figure 4. When at RT-600 °C, the air relative humidity decreased with the increasing temperature, leading to the water film adsorbed on the wear tracks was broken[10]. That caused the severe wear in the coatings and the Al₂O₃ balls.

![Figure 3](image_url)

**Figure 3** Function curves of friction coefficient and time of TiAgN-Ag coatings at different temperatures

The friction coefficient was lower at 600 °C-300 °C. After running-in stage of 2 min, the friction coefficient was stably at 0.6, which was attributed to the lubrication of Ag and oxides. In the beginning, the temperature of wear tracks area was higher, Ag atoms diffused to the surface and acted as lubricant. As the ambient temperature decreased with time, the lubricant effect of Ag decreased. At the same time, more and more oxides generated during wear process, such as TiO₂ and Al₂O₃, which can also act as lubrication. As a result, the TiAgN-Ag coating kept a lower friction coefficient during abrasion at the temperature from 600 °C decreased to 300 °C and showed better wear resistance. The wear rate of the TiAgN-Ag coating at the temperature from 600 °C decreased to 300 °C was 0.085×10⁻³mm³/(Nm).

Incertiet.al[11] prepared CrN-Ag film by means of magnetron sputtering and PVD reactive arc discharge evaporation. They found annealing treatment of the coatings lead to coarsening of superficial Ag clusters and segregation out of the CrN matrix. The coatings showed the lowest coefficient of friction appeared at 600 °C, no matter which technology was used. The excellent wear resistance was related to continuous Ag segregation out of CrN matrix, which enables self-lubrication. Kutschejet.al[12] also reported the similar tribological properties of CrN-Ag coatings.
4. Conclusions

(1) TiAlN-Ag composite coatings were deposited by magnetron sputtering. The coatings mainly consisted of TiAlN phases with (111) preferred orientation.

(2) When at RT-600 °C, the severe wear occurred between coatings and Al$_2$O$_3$ balls resulted from the lower relative humidity. Ag and oxides acted as lubricants at 600 °C-300 °C, which lead to the lower friction coefficient of 0.6 and lower wear rate of $0.085 \times 10^{-3} \text{mm}^3/(\text{Nm})$.

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