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Tribological properties of plasma sprayed NiAl-Ag-Ta-Cr_2O_3 composite lubrication coatings from room temperature to 750°C

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Abstract. With the rapid advances in science and technology, the core parts and techniques in tribosystems rely on solid-lubricating materials at wide temperature ranges for durability, especially for designing and producing materials with low friction coefficient and high wear resistance over wide temperature ranges. In this paper, a series of NiAl-Ag-Ta-Cr_2O_3 composite coatings with different contents of Ta and Ag were deposited by plasma spraying, and their tribological properties at RT-750°C under dry sliding conditions were investigated by a ball-on-disk tribometer. The friction products and compositions of worn surfaces at different temperatures were investigated. The deposited NiAl-Ag-Ta-Cr_2O_3 composite coatings exhibited excellent wear resistance at RT (10^{-7} mm^3/N m), while the friction coefficients were lower than 0.5 at RT to 750°C. The contents of Ta and Ag determined the mechanical properties of coatings and greatly affected the tribological properties by creating a tribo-film (lubricant film and glaze layer). NiAl-10Ag-5Ta-20Cr_2O_3 coating shows better tribological properties under all temperatures, while the wear rate is reduced to $5.58 \cdot 10^{-6}$ mm^3/N m at 750°C, and the friction coefficient falls to 0.21 at 600°C. Excellent lubrication and wear resistance of composite coatings at high temperatures is mainly attributed to the Ag acting as the lubrication phase, and the top surface of the wear track being covered with a smooth glaze layer, which consisted of Ag, Ta_2O_5, Cr_2O_3, and $AgTaO_3$. In addition, the lubrication mechanism of silver tantalate was also briefly discussed.

Keywords: lubricating coatings; plasma spraying; silver tantalate; wide temperature range.

1. INTRODUCTION

In modern high-tech industries, some mechanical parts frequently experience extremely harsh working conditions, like high/low temperature (wide-temperature ranges). The lubrication and anti-wear of relative sliding parts are dominant for the reliability and life expectancy of the whole system. So new lubricating materials with continuous lubrication under a wide temperature range must be urgently developed [1–3]. The developed PS400 coating by NASA consisted of 70 wt.% Ni-Mo-Al as the main matrix, 20‘wt.% Cr_2O_3 as reinforcement phase, and the lubricating phase (5 wt.% Ag and 5‘wt.% CaF_2/BaF_2) was reduced to 10 wt.% compared to the PS304 with a 20 wt.%. It was also beneficial to improve the surface quality and high-temperature strength. The research on solid lubricating composite coatings tended to reduce the content of solid lubricants to improve their strength [4, 5]. CaF_2/CaF_2, which is a eutectic compound with a low melting point, plays the role of lubrication at elevated temperatures and inevitably debases the strength of coatings.

A lot of research reported that some other solid lubricants could replace eutectic fluoride. Chen *et al.* [6, 7] investigated the tribological properties of NiMoAl-Ag and NiMoAl-Cr_3C_2-Ag coating at elevated temperatures. The formed ternary oxide (Ag_2MoO_4) enamel layer on the worn surface showed protective and lubricative properties at 600°C, being able to partially substitute eutectic fluoride. Du *et at*. [8] prepared NiCoCrAlY-Cr_2O_3-Ag-Mo composite coating consisting of 10 wt% Ag-Mo by air plasma spraying technology, with a friction coefficient of 0.3–0.8 at the temperature range from RT to 800°C against Si_3N_4, especially under 800°C, the formation of Ag_2MoO_4 resulted in a relatively low wear rate (10^{-6} mm^3/Nm). For NiCoCrAlY-Cr_2O_3-$AgVO_4$ coating [9], the friction coefficient was 0.20–0.55 from RT to 800°C, while the wear rates were at an order of 10^{-4} mm^3/Nm. As a reliable solid lubricant, silver was usually used in hard coatings to improve their tribological properties, mostly presented in the form of Ag cluster or Ag-based transition metal oxides (i.e., $Ag_xTM_yO_z$) [10]. $Ag_xTM_yO_z$ was used to improve the tribological properties of hard coatings at high temperatures [11], and it was considered to fill the blank of the solid lubricant at 800~1000°C [12–14]. Silver tantalate ($AgTaO_3$) can be formed on the frictional surface at high temperatures above 600°C [15]. The prepared $AgTaO_3$ coating shows a friction coefficient of 0.06 at 750°C due to the naturally occurring $AgTaO_3$, and Ta_2O_5 [16, 17]. Gao *et al.* [18, 19] investigated the sliding resistance of $AgTaO_3$ at different temperatures by MD simulation and experimental methods. It showed that the lowest friction forces were achieved at 750°C, good according to the results, while the formed tribo-film of Ag clusters and surrounding Ta_2O_5 during high temperature sliding.

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The research on composite coatings with Ta and Ag as additives by plasma spraying technology will help extend the application of Ta and increase the categories of lubricating material. In this study, four NiAl-Ag-Ta-Cr_2O_3 composite coatings with different contents of Ag and Ta were prepared by plasma spraying technology, while the NiAl acted as the matrix phase, and Cr_2O_3 as the reinforcement phase. The tribological properties of coatings at RT to 750°C were tested, the effect of Ag and Ta concentration on the properties was investigated, and the lubricating and wear resistance mechanism of the composite coatings at high temperatures was briefly discussed.

2. EXPERIMENT DETAILS

2.1. Materials and methods

Al-coated Ni and Cr_2O_3 powders were purchased from Yong Xing Lian Nonferrous Metals Co. Ltd (Beijing), while the Al-coated Ni powder comprised of 95 wt.% Ni and 5 wt.% Al. Ag and Ta powders were provided by ZhongNuo Material. Ltd (Beijing). The mass fraction of powders is shown in Table 1. Composite coatings presented excellent tribology capability when the contents of the reinforcement phase were 17–22 wt.%. In this study, the content of Cr_2O_3 was set as 20 wt.%. The mixed powder (Ta-Ag-Cr_2O_3) was prepared by sintering and breaking with the compositions in Table 1. The original powders were blended in an XGB2 planetary ball mill for 8 hours with a ratio of powder: ball (Si_3N_4): alcohol = 1:3:1, and the speed was 300 r/min. Then they dried under 80°C for 3 hours and sintered at 90°C in a vacuum of 10^{-3} Pa for 3 hours, after which the powders were screened by 200~300 mesh. The Al-coated Ni powders were mechanically blended with Ta-Ag-Cr_2O_3 powders and dried for 4 hours at 60°C. Carbon steel was chosen as substrate, and it was sandblasted and cleaned before depositing, then the NiAl-Ag-Ta-Cr_2O_3 composite coatings were deposited by using a DH-1080 atmospheric plasma spray system. The parameters of the plasma spraying process are shown in Table 2.

Table 1
As-sprayed power

Number	NiAl	Ag	Ta	Cr_2O_3	Class
1	70	5	5	20	5A5T
2	65	10	5	20	10A5T
3	60	10	10	20	10A10T
4	60	15	5	20	15A5T
5	80	0	0	20	0A0T

Table 2
Plasma spraying parameters

Items	Value
Current	690 A
Voltage	28–32 V
Spray distance	100 mm
Argon flow rate	3000 L/h
Hydrogen flow rate	120 L/h
Powder feed rate	1 Kg/h
Powder gas flow rate	0.4 m^3/h
Spray gun walking speed	15 mm/s

2.2. Friction and wear test

A ball-on-disk high-temperature tribometer was carried out to test the tribological properties of coatings. A Si_3N_4 (Ra = 9.4 nm) ball with a diameter of 6 mm was chosen as the upper specimen due to its high hardness and chemical inertness. Before friction tests, the prepared coatings were mechanically grounded with 600 grit emery paper, and ultrasonic cleaned for 10 minutes. The experiment temperatures were room temperature (RT), 200, 400, 600, and 750°C. The rotation speed was 300 r/min (0.157 m/s), and each test was sustained for 60 min (565.2 m, 18000 r) under a 5 N load with a 5 mm radius. During the test, the friction coefficients were continually recorded by the software, and the wear rates were calculated by $W = V/SF$, where V was the wear volume loss in mm^3 from the measured sectional profile of the wear track, S was the total sliding distance in m and F was the applied load in N.

2.3. Characterization

The morphologies of as-sprayed powders, prepared coatings, and worn surfaces were observed by scanning electron microscope (SEM, Quanta 250F, FEI, America). The phase composition of powders and deposited coatings was determined by X-ray diffractometer (XRD, D8, BRUKER, Germany) using Cu Kα radiation. HVS-1000-Z was carried out to test the microhardness with a 2 N load for 10 s. After the tribological test, the morphologies of worn surfaces were observed by Laser Scanning Confocal Microscope and SEM equipped with an energy dispersive spectrometer (EDS), in addition, the variations of the phase compositions on worn surfaces were investigated by Raman spectrometer (HORIBA, Japan) with an excitation wavelength of 532 nm.

3. RESULTS AND DISCUSSION

3.1. Characterization of coating and powder

The aggregated particles of Ag-Ta-Cr_2O_3 composite powder are shown in Fig. 1a. After mechanical alloying, sintering, and crushing, the combined particles were of different sizes, with a size distribution range of 10–50 μm, which is suitable for spraying. Figure1b shows the morphologies of Al-coated Ni powder.

To identify the composition of the synthesized powder and the deposited coating. Figure 2 shows the XRD diffraction pattern of NiAl-10Ag-5Ta-Cr_2O_3 coating and powder. No characteristic peaks of oxide or new phase can be observed from the pattern of the powder, indicating the powder was less oxidized and without other chemical reactions triggered. The composite coating is

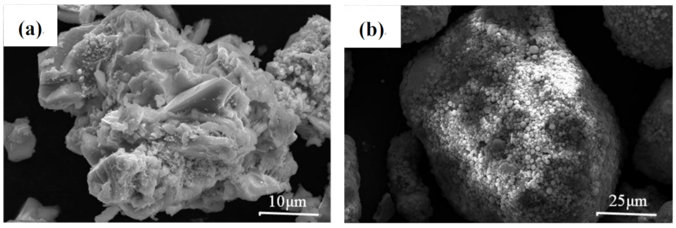

Fig. 1. SEM morphology of feedstock powders: (a) Ag-Ta-Cr_2O_3 composite powder and (b) Al-coated Ni

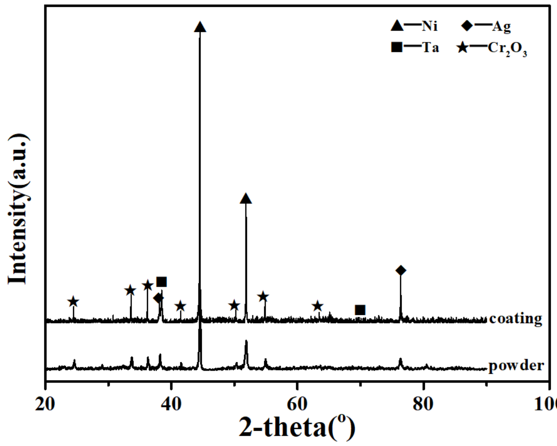

Fig. 2. XRD patterns of NiAl-10Ag-5Ta-Cr_2O_3 coating and powder

mainly composed of Ni and Ni-based solid solutions. The peaks of Ag and Ta existing in composite coating demonstrate that the Ag and Ta do not alloy in the matrix during the plasma spraying process. Further, the peaks of Cr_2O_3 are relatively obvious, demonstrating that all the powders were successfully deposited on the substrate. Furthermore, no other new phases are detected in the deposited coating, which means the feedstock powders do not experience obvious oxidation or decomposition during deposition.

Figure 3a shows the cross-section morphology of NiAl-10Ag-5Ta-Cr_2O_3 coating, a typical lamellar structure with a thickness of about 300 μm, consisting of ribbon-like layers, and tightly combined each other with fewer pores or cracks. Benefiting from the suitable deposition conditions and the reaction of Al-coated Ni power, the interface between substrate and coating shows fewer cracks. Through the element distribution test, the bright and white areas mainly consist of Ni and Al, and the black area mainly consists of Cr_2O_3 with stripy distribution. The heterogeneous distribution of the compositions of deposited coatings may lead to the maldistribution of Vickers hardness values. For this reason, the microhardness of the section was conducted at 40 μm intervals to evaluate the mechanical properties of composite coatings. As shown in Fig. 3b, the microhardness of NiAl-Cr_2O_3 coating (595 HV) is much higher than NiAl coating (180 HV) due to the reinforcement of Cr_2O_3. The composite coatings containing soft metal (Ag and Ta) show a lower microhardness. The microhardness of 15A5T coating significantly decreases to 350 HV due to the higher sliver contents (15 wt.%).

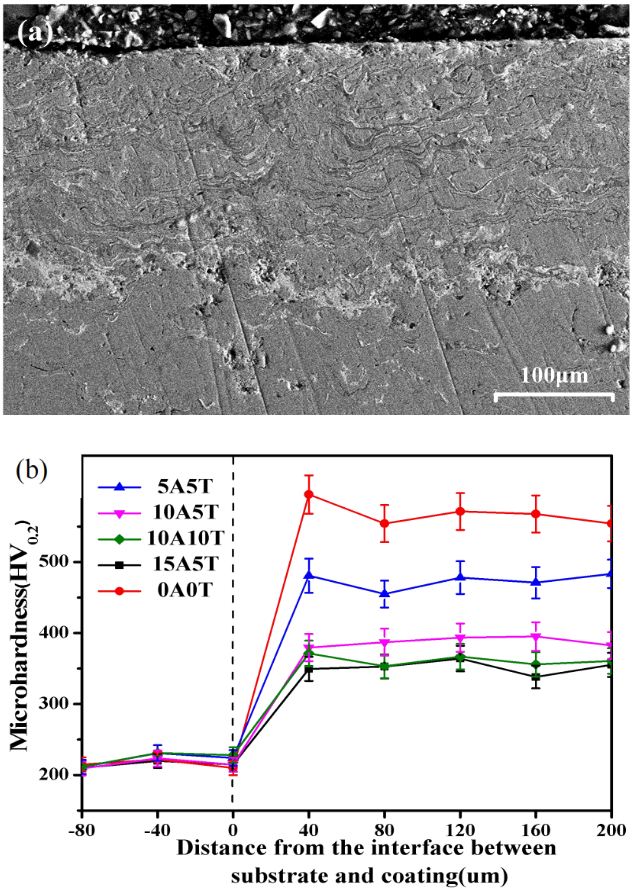

Fig. 3. (a) SEM images and (b) microhardness of cross-section of NiAl-Ag-Ta-Cr_2O_3 coatings

3.2. Tribological properties

Figure 4a further gives the Cof curves of 10A5T coating at RT to 750°C, with the Cof relatively stable as sliding distance increases and sustained in a relatively lower value.

Figure 4b shows the average coefficient of friction (Cof) of NiAl-Ag-Ta-Cr_2O_3 coatings sliding against Si_3N_4 ball from RT to 750°C. Obviously, the Cof decreases from 0.5 to 0.2–0.3 with the temperature increasing from RT to 600°C and then increases at 750°C. The 5A5T coating with lower contents of Ta and Ag shows the highest Cof values at all temperatures due to the insufficient lubrication phase. As the contents of Ag increase to 10 wt.%, the Cof of 10A5T coating slightly decreases at RT and 200°C, and significantly decreases at 400, 600, and 750°C (0.32, 0.21, 0.25). The Cof of 10A10T coating increases at high temperatures (0.32–0.38, 0.21–0.26, 0.25–0.28). The Cof of 15A5T coating with a higher Ag is only lower than the 5A5T coating at all temperatures, this may be due to the excessive lubricants leading to decreased mechanical properties (473–351 HV). The Cof of all coatings is not much different at RT and 200°C compared with each other, and the 10A5T coating shows a lower value at all temperatures, especially at 600°C (0.22).

The wear rates of composite coatings, NiAl-Cr_2O_3, and substrate at RT to 750°C were shown in Fig. 5a, 5b. Apparently, the wear rate of NiAl-Ag-Ta-Cr_2O_3 coatings is much lower than

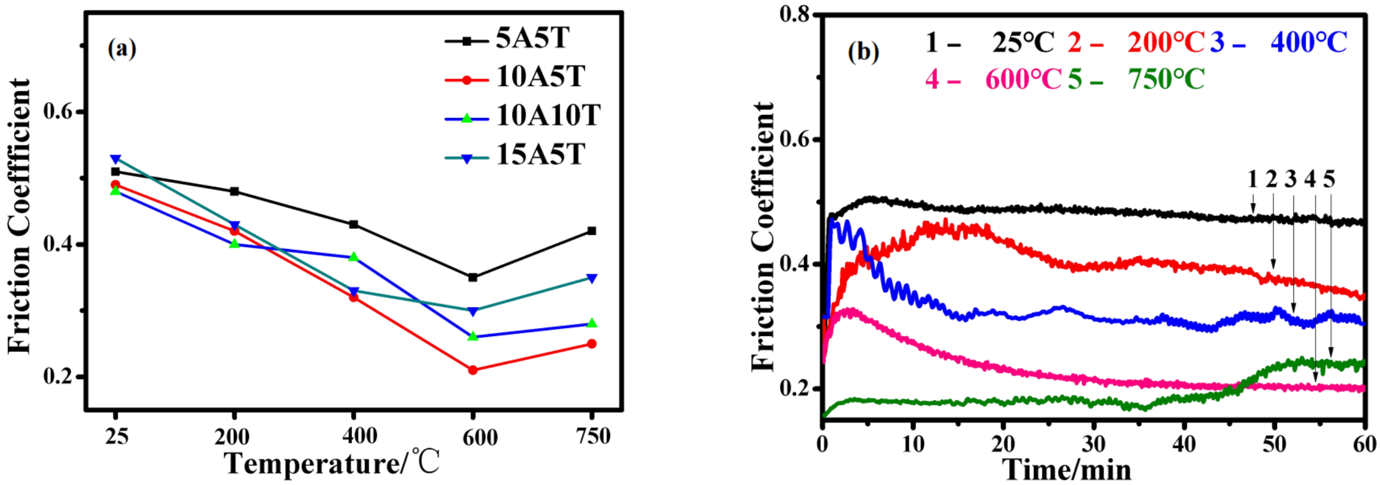

Fig. 4. (a) Friction coefficient of NiAl-Ag-Ta-Cr_2O_3 composite coatings within 1 hour at 25, 200, 400, 600, and 750°C, respectively; (b) Friction coefficient of 10A5T coating within 1 hour at 25, 200, 400, 600 and 750°C, respectively

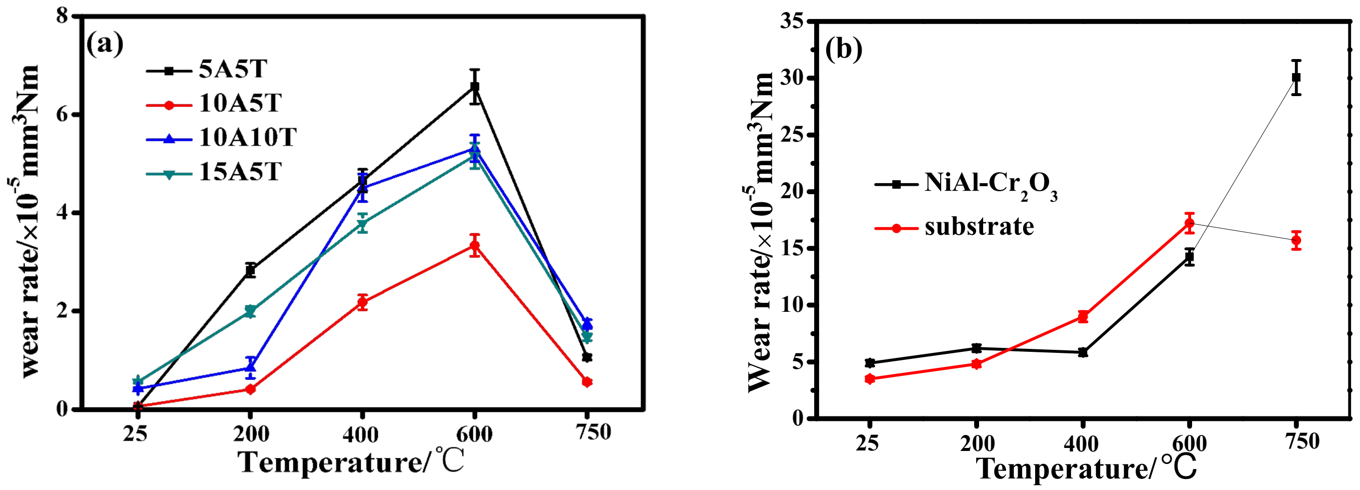

Fig. 5. (a) Wear rate of NiAl-Ag-Ta-Cr_2O_3 coatings at 25, 200, 400, 600 and 750°C, respectively; and (b) Wear rate of NiAl-Cr_2O_3 and substrate at 25, 200, 400, 600 and 750°C, respectively

the NiAl-Cr_2O_3 at all temperatures, while it indicates that the added Ta and Ag can effectively improve the wear resistance of coatings at a wide temperature. With increasing test temperature, all the deposited NiAl-Ag-Ta-Cr_2O_3 coatings show an increased wear rate, and achieve a maximum at 600°C, then decline sharply at 750°C. At RT, the 15A5T and 10A5T coating shows the lowest wear rate (10^{-7} mm^3/Nm orders of magnitude). At 200°C, the wear rate of 10A5T and 10A10T coating ($4.07 \cdot 10^{-6}$ mm^3/Nm, $8.45 \cdot 10^{-6}$ mm^3/Nm) is lower than that of 5A5T and 15A5T coating. The 10A5T coating has the lowest wear rate ($2.18 \cdot 10^{-5}$ mm^3/Nm) at 400°C, this may be due to the appropriate contents of lubricant and relatively high H. At 600°C, the wear rate of NiAl-Ag-Ta-Cr_2O_3 coatings increases, and the 10A5T coating still shows the lowest wear rate ($3.336 \cdot 10^{-5}$ mm^3/Nm). Unexpectedly, the wear rates (1.057, 0.558, 1.735, $1.473 \cdot 10^{-5}$ mm^3/Nm) sharply decrease at 750°C, and the 10A5T coating is reduced to a magnitude of 10^{-6} mm^3/Nm.

3.3. Analysis of worn surface

To figure out the abrasion wear form under different temperatures, the influence of temperature on the morphologies of worn surface was discussed. Figures 6a–6e show the three-dimensional topographies of 10A5T coating worn surface at different temperatures, the shallow worn surface at RT increases with the test temperature rising, the deepest worn surface is obtained at 600°C and sharply decreases at 750°C. Besides, the worn surface is much smoother and lighter, and it indicates that Ag and Ta play an important role in the wear reduction at this temperature. In addition, the worn surface SEM images of NiAl-10Ag-5Ta-Cr_2O_3 coating at RT to 600°C are shown in Fig. 7. The composite coating shows the characteristics of fatigue cracking, pitting, and furrow at RT, revealing that the wear mechanism is typical abrasive wear. It is more serious at 200°C, some unexfoliated lamellar patches can be observed due to the delamination. The worn surface was much smoother at 400°C. As shown in Fig. 7d, the worn surface shows ev-

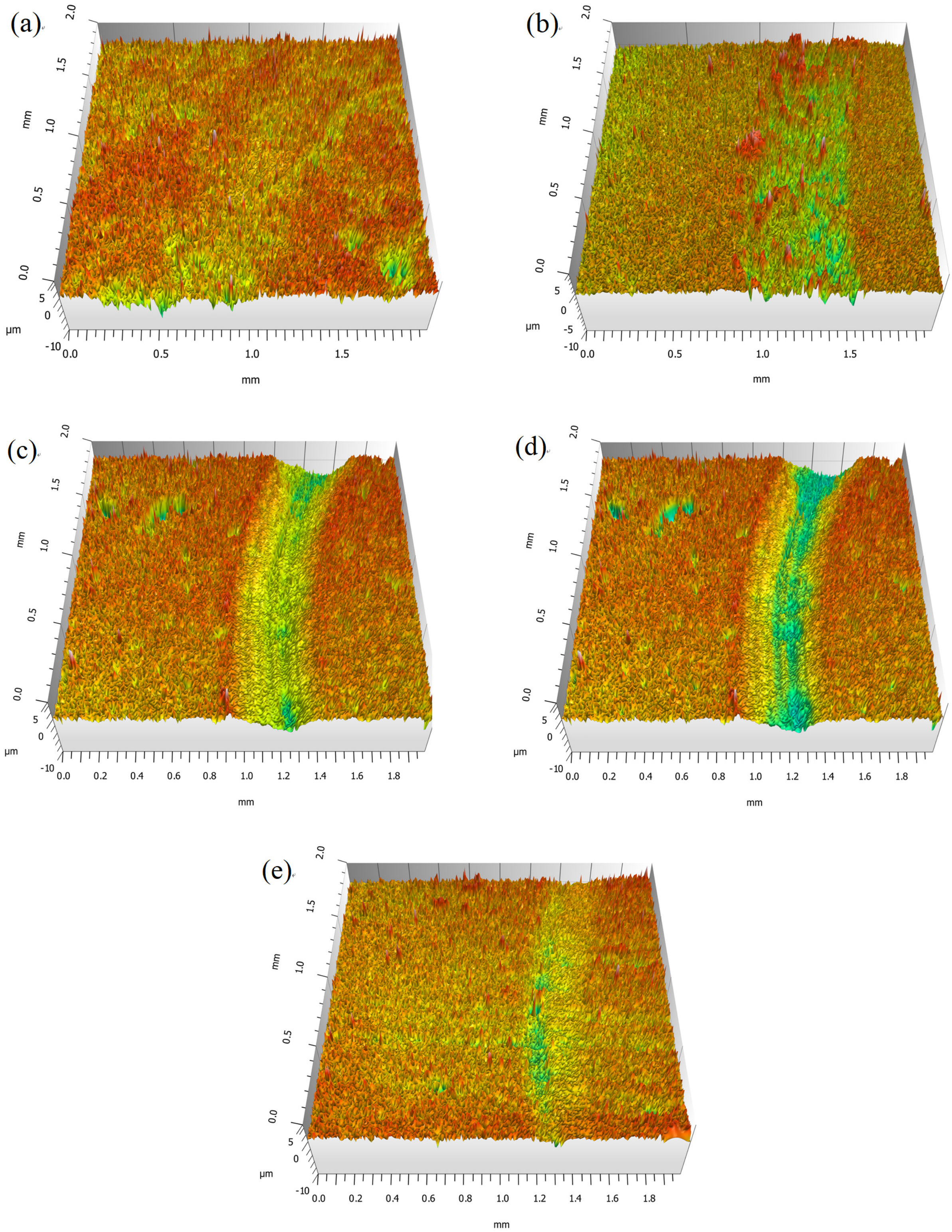

Fig. 6. Three-dimensional images of worn surface for 10A5T coating at different temperatures: (a), (b), (c), (d), and (e), respectively corresponding 25, 200, 400, 600, and 750°C

ident adhesion wear and plastic deformation at 600°C, while the composite coating suffers the most serious abrasion at this temperature. The wear rates of all composite coating greatly decreased at 750°C. The SEM images of the worn surface at 750°C are shown in Fig. 8. The worn surface becomes much smoother compared with other samples at RT-600°C. The abrasive wear and adhesive wear are effectively suppressed at this temperature, a relatively continuous and dense glaze film is formed

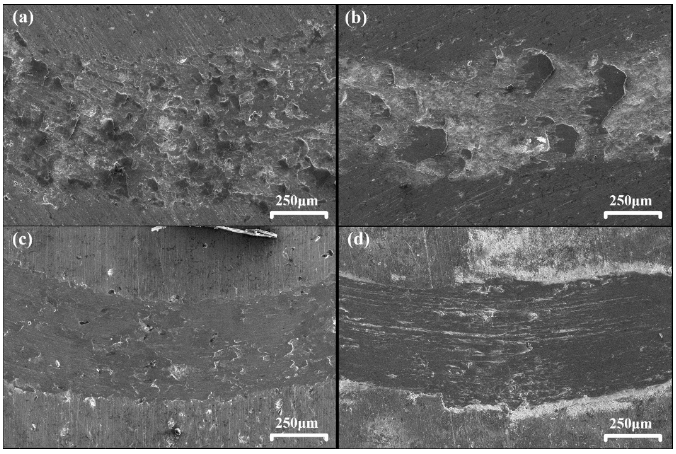

Fig. 7. SEM images of the worn surface of 10A5T coating at a different temperature: (a), (b), (c), and (d), respectively corresponding to 25, 200, 400, and 600°C

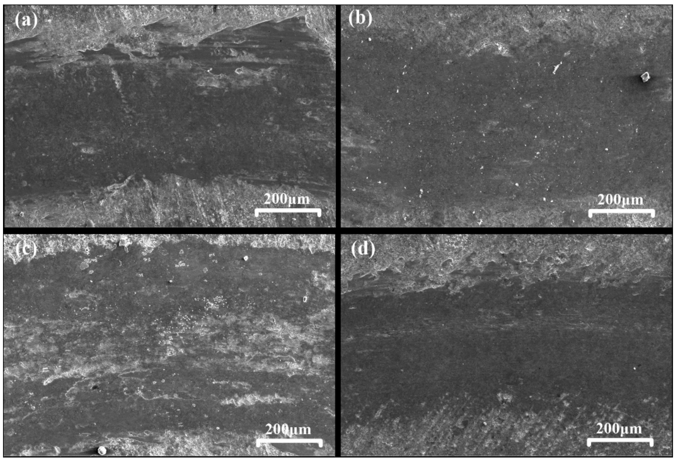

Fig. 8. SEM images of the worn surface of composite coatings at 750°C: (a), (b), (c), and (d), respectively, corresponding to 5A5T, 10A5T, 10A10T, and 15A5T coating

after friction testing at high temperatures, and the glaze layer is mainly responsible for the low friction coefficient and wear rate. Compared with each other, the 10A5T and 15A5T coating shows a higher continuity and completeness friction layer, while corresponding to the lower wear rate.

3.4. Discussion

To figure out the lubrication and wear resistance of NiAl-Ag-Ta-Cr_2O_3 composite coatings, 10A5T coating was selected as the research object, and its worn surface compositions under different temperatures were analyzed by EDS and Raman. Table 3 shows the elements of worn and unworn surfaces at 400, 600, and 750°C. Figure 9 shows the corresponding Raman spectrograms. In Fig. 9a, the weak characteristic peaks of $AgTaO_3$ and Ta_2O_5 are presented in areas A and B, and the much stronger characteristic peaks of Ta_2O_5 exist in areas C and D. Figure 9b indicates that the worn surface mainly consists of the discontinuous oxide layer and uncovered Cr_2O_3 and Ni. At 600°C, a large amount of oxides forms due to enhanced oxidation reactions, the obvious characteristic peaks of $AgTaO_3$ appear at 700 cm^{-1} in Raman spectra, indicating that the tantalum oxides mainly exist in the form of $AgTaO_3$ and Ta_2O_5, while the wear rate is the highest at this temperature. As we know, in oxidation wear, volume loss mainly comes from the wear of the oxide layer. It is shown that delamination of the oxide layer takes place due to internal stress when it reaches a critical thickness [20], due to the formed oxide film not being dense and attached not well. Volume loss mainly comes from the oxide layer at this temperature.

Table 3
Element contents of the worn and unworn surface of 10A5T coating at 400, 600, and 750°C

Element (wt. %)	400°C unworn	400°C worn	600°C unworn	600°C worn	750°C unworn	750°C worn
O	27.9	35.5	30.8	35.9	34.9	33.3
Al	3.2	1.7	2	1.2	2.6	1.3
Cr	19.2	20.6	21.3	16.8	17.1	18.8
Ni	41.3	30.5	33.7	26.3	28.9	20.4
Ag	3.7	7.3	6.8	13.7	7.4	14.1
Ta	4.7	4.4	5.4	6.1	9.1	12.1
total	100.00	100.00	100.00	100.00	100.00	100.00

As seen in Fig. 8b and Fig. 9e, a more continuous and relatively complete glaze film forms after the friction test at 750°C, which is responsible for the increased wear resistance. The EDS

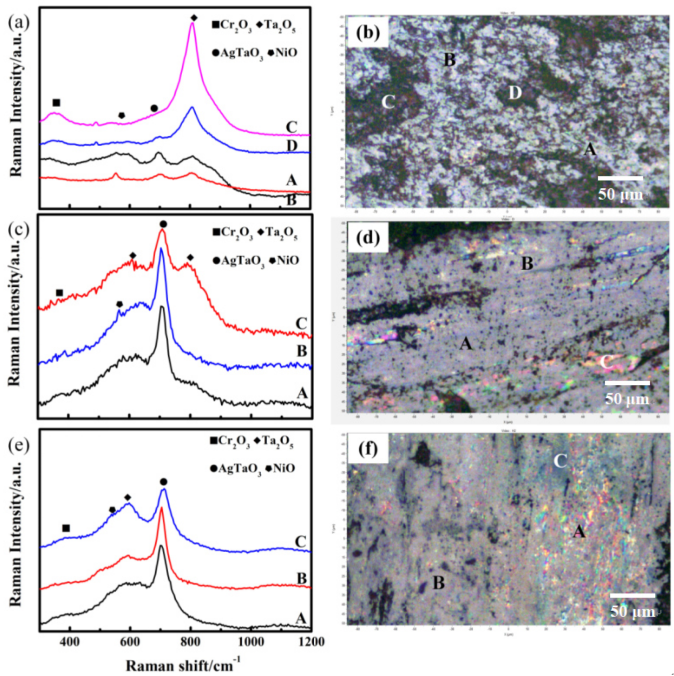

Fig. 9. Raman spectrogram of worn surface of 10A5T coating at different temperatures: (a), (c), and (e), respectively, corresponding to 400, 600, and 750°C, (b), (d), and (f) corresponding optical image

results (Table 3) show that the Ag and Ta contents are higher in the worn regions compared to the unworn surfaces and other temperature regions for the glaze layer. This is mainly due to the lubricant Ag with low shear stress and easiness of being smeared on the worn surface during sliding. In addition, the Raman intensity of $AgTaO_3$ and Ta_2O_5 is much stronger, and as we know, $AgTaO_3$ has a perovskite cubic structure, which can be produced during friction above 507°C, with the melting point of $AgTaO_3$ at 1185°C [15]. The friction coefficient of pure $AgTaO_3$ coating is 0.06 at 400°C and the friction coefficient of TaN-Ag coating is 0.23 at 750°C [16–18]. The formed friction-induced oxidation and high temperatures oxidation (Ta_2O_5, Cr_2O_3, and NiO, etc.) provided wear resistance as a support phase. Thus, the Ag-rich glaze layers played a key role in decreasing the friction coefficient and wear rate. On top of that, according to the SEM images (Figs. 7, 8, and 9), EDS results (Table 3), and Raman (Fig. 9), at low temperatures (RT-200°C), the wear rate of composite coatings has no obvious difference, indicates the lubricate (Ag) and good mechanical properties of matrix are responsible for the antifriction and wear resistance. At the middle temperature (400–600°C), volume loss mainly comes from the oxide layer, the appropriate contents of lubricant and better mechanical properties can effectively reduce the wear rate and friction coefficient for NiAl-10Ag-5Ta-Cr_2O_3 coating. At higher temperatures (750°C), the tribological properties are improved by the tribochemical products and oxides, the relatively continuous distribution glaze layers on the worn surface mainly consists of $AgTaO_3$ Cr_2O_3, Ta_2O_5, and Ag, which plays a crucial role in improving the anti-wear and friction reducing abilities at high temperature.

4. CONCLUSIONS

NiAl-Ag-Ta-Cr_2O_3 coatings with different contents of Ta and Ag were successfully deposited on the carbon steel surface. The friction coefficient of NiAl-10Ag-5Ta-Cr_2O_3 coating was lower than 0.5 from RT to 750°C.

The lowest friction coefficient (0.21) was achieved at 600°C, while the lowest wear rate in the order of 10^{-7} mm^3/Nm was obtained at RT, the wear resistance and lubrication of the coating mainly rely on Cr_2O_3 and Ag at the temperature below 400°C.

The wear rate at 750°C was prominent ($5.58 \cdot 10^{-6}$ mm^3/Nm), the composited glaze layers which mainly consisted of $AgTaO_3$ Cr_2O_3, Ta_2O_5, and Ag are proposed to be the main reasons for reducing friction coefficient and anti-wear at 750°C.

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