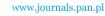
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# Tribological properties of plasma sprayed NiAl-Ag-Ta-Cr<sub>2</sub>O<sub>3</sub> composite lubrication coatings from room temperature to 750 °C

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**Abstract:** With the rapid advances in science and technology, the core parts and techniques in tribo-systems rely on the solid-lubricating materials at wide-temperature range for durability, especially for designing and producing materials with low friction coefficient and high wear resistance over a wide temperature ranges. In this paper, a series of NiAl-Ag-Ta-Cr<sub>2</sub>O<sub>3</sub> composite coatings with different contents of Ta and Ag were deposited by plasma spraying, 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 surface at different temperature were investigated. The deposited NiAl-Ag-Ta-Cr<sub>2</sub>O<sub>3</sub> composite coatings exhibited excellent wear resistance at RT ( $10^{-7}$  mm<sup>3</sup>/N m), while the friction coefficients lower than 0.5 at RT to 750 °C. The contents of Ta and Ag decided mechanical properties of coatings, and greatly affected the tribological properties by creating an tribo-film (lubricant film and glaze layer). NiAl-10Ag-5Ta-20Cr<sub>2</sub>O<sub>3</sub> coating shows better tribological properties over all temperature, while the wear rate reduces to  $5.58 \times 10^{-6}$  mm<sup>3</sup>/N m at 750 °C, and the friction coefficient reduces to 0.21 at 600 °C. The excellent lubrication and wear resistance of composite coatings at high temperatures are mainly attributed to the Ag acted as lubrication phase, and the top surface of wear track was covered with a smooth glaze layer, which consisted of Ag, Ta<sub>2</sub>O<sub>5</sub>, Cr<sub>2</sub>O<sub>3</sub> and AgTaO<sub>3</sub>. In addition, the lubrication mechanism of silver tantalate was also briefly discussed.

Key words: lubricating coatings; plasma spraying; silver tantalate; wide temperature range

# **1. INTRODUCTION**

In modern high-tech industries, some mechanical parts frequently experienced extremely harsh working conditions, like high/low temperature (wide-temperature ranges). The lubrication and anti-wear of relative sliding parts is dominant for the reliability and life expectancy of the wpore system. So the new lubricating materials with continuous lubrication over a wide temperature ranges are urgent to be developed [1-3]. The developed PS400 coating by NASA was consisted of 70 wt.% Ni-Mo-Al as main matrix, 20 wt.% Cr2O3 as reinforcement phase, and the lubricating phase (5 wt.% Ag and 5 wt.% CaF<sub>2</sub>/BaF<sub>2</sub>) reduce to 10 wt.% compare the PS304 with a 20 wt.%, while it is beneficial to improve the surface quality and high temperature strength. And the researches of solid lubricating composite coatings tended to reduce the content of solid lubricant to improve their strength [4-5]. CaF<sub>2</sub>/CaF<sub>2</sub> is eutectic compound with a low melting point, it plays the role of lubrication at elevated temperature and inevitably debases the strength of coatings.

Many researches has been reported that some other solid lubricants could take place the eutectic fluoride. Chen et.al [6-7] investigated the tribological properties of NiMoAl-Ag and NiMoAl-Cr<sub>3</sub>C<sub>2</sub>-Ag coating at elevated temperatures, the formed ternary oxide (Ag<sub>2</sub>MoO<sub>4</sub>) enamel layer on the worn surface show a protective and lubrication at 600 °C, while it can partially substitute the role of eutectic fluoride. Du et.al [8] prepared NiCoCrAlY-Cr<sub>2</sub>O<sub>3</sub>-Ag-Mo composite coating with consisting of 10 wt% Ag-Mo by air plasma spraying technology, while it shows a friction coefficient of 0.3-0.8 at the temperature range from RT to 800 °C against Si<sub>3</sub>N<sub>4</sub>, especially under 800 °C, the formation of Ag<sub>2</sub>MoO<sub>4</sub> resulted a relatively low wear rate (10<sup>-6</sup> mm<sup>3</sup>/Nm). For NiCoCrAlY-Cr<sub>2</sub>O<sub>3</sub>-AgVO<sub>4</sub> 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<sup>-4</sup> mm<sup>3</sup>/Nm. Silver as a reliable solid lubricant usually was used in hard coatings to improve their





tribological properties, mostly it 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 temperature [11], it was considered to fill the blank of the solid lubricant at 800~1000 °C [12-14]. Silver tantalate (AgTaO<sub>3</sub>) can be formed on the frictional surface at high temperature above 600 °C [15]. The prepared AgTaO<sub>3</sub> coating shows a friction coefficient of 0.06 at 750 °C due to the naturally occurred AgTaO<sub>3</sub>, Ta<sub>2</sub>O<sub>5</sub> [16-17]. Gao et.al [18-19] investigated the sliding resistance of AgTaO<sub>3</sub> at different temperatures by MD simulation and experimental methods. It showed that the lowest friction forces were achieved at 750 °C, good in according with the results, while the formed tribo-film of Ag clusters and surrounding Ta<sub>2</sub>O<sub>5</sub> during high temperature sliding.

The researches of composite coatings with Ta and Ag as additive by plasma spraying technology will be helpful for extending the application of Ta and increasing the categories of lubricating material. In this study, four NiAl-Ag-Ta-Cr<sub>2</sub>O<sub>3</sub> composite coatings with different contents of Ag and Ta were prepared by plasma spraying technology, while the NiAl acted as matrix phase,  $Cr_2O_3$  acted as reinforcement phase. The tribological properties of coatings at RT to 750 °C were tested, the effect of Ag and Ta concentration on the properties were investigated, and the lubricating and wear resistance mechanism of the composite coatings at high temperature was briefly discussed.

#### 2. EXPERIMENT DETAILS

#### 2.1. Materials and methods

Al coated Ni and Cr<sub>2</sub>O<sub>3</sub> powders were purchased from Yong Xing Lian Nonferrous Metals Co. Ltd (Beijing), while the Al coated Ni powder was 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 was shown in Table 1. Composite coatings presented excellent tribology capability when the contents of reinforcement phase are 17-22 wt.%. In this study, the content of Cr<sub>2</sub>O<sub>3</sub> was set as 20 wt.%. The mixed powder (Ta-Ag-Cr<sub>2</sub>O<sub>3</sub>) was prepared by sintering and breaking with the compositions in Table. 1. The original powders were blended in a XGB2 planetary ball mill for 8h with a ratio of powder: ball  $(Si_3N_4)$ : alcohol = 1:3:1, and the speed was 300 r/min. Then dried under 80 °C for 3 h and sintered at 900 °C under a vacuum of 10<sup>-3</sup> Pa for 3 h, after that, the powders were screened by 200~300 mesh. The Al coated Ni powders were mechanical blended with Ta-Ag-Cr<sub>2</sub>O<sub>3</sub> powders and dried for 4h at 60 °C. Choose carbon steel as substrate, while it was sand blasted and cleaned before depositing, then the NiAl-Ag-Ta-Cr<sub>2</sub>O<sub>3</sub> composite coatings were deposited by using DH-1080 atmospheric plasma spray system. The parameters of plasma spraying process were shown in Table. 2.

 TABLE 1. as-sprayed power

Number	NiAl	Ag	Та	$Cr_2O_3$	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									
	Ite	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 <sup>3</sup> /h					

15 mm/s

#### 2.2. Friction and wear test

Spray gun walking speed

Ball on disk high temperature tribometer was carried out to test the tribological properties of coatings,  $Si_3N_4$  (Ra=9.4 nm) ball with a diameter 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 rotate speed was 300 r/min (0.157 m/s), each test sustained for 60 min (565.2 m, 18000 r) under 5 N load with a 5 mm radius. During 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<sup>3</sup> from the measured sectional profile of 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 surface were observed by scanning electron microscope (SEM, Quanta 250F, FEI, America). The phases composition of powders and deposited coatings were determined by X-ray diffractometer (XRD, D8, BRUKER, Germany) using Cu K $\alpha$  radiation. HVS-1000-Z was carried out to test the micro-hardness with a 2 N load for 10 s. After 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<sub>2</sub>O<sub>3</sub> composite powder shows in Fig. 1(a). After mechanical alloying, sintering and crushing, the particles with different size combined together, with a size distribution range of 10-50  $\mu$ m, which is suitable for spraying. Fig. 1(b) shows the morphologies of Al-coated Ni powder.



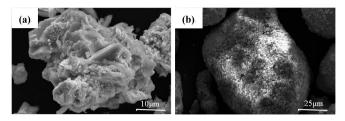


Fig. 1. SEM morphology of feedstock powders: (a) Ag-Ta- $Cr_2O_3$  composite powder and (b) Al-coated Ni

To identify the composition of the synthesized powder and the deposited coating. Fig. 2 shows the XRD diffraction pattern of NiAl-10Ag-5Ta-Cr<sub>2</sub>O<sub>3</sub> coating and powder. No characteristic peaks of oxide or new phase can be observed from the pattern of powder, indicates the powder was less oxidized and without other chemical reaction triggered. The composite coating mainly composed of Ni and Ni based solid solution. The peaks of Ag and Ta exist in composite coating demonstrate that the Ag and Ta did not alloyed in matrix during plasma spraying process, further the peaks of Cr<sub>2</sub>O<sub>3</sub> is relatively obvious, demonstrate well that all the powders have been successfully deposited on the substrate. Furthermore, no other new phases are detected in the deposited coating, it means the feedstock powders without experiencing obvious oxidation or decomposition during deposition.

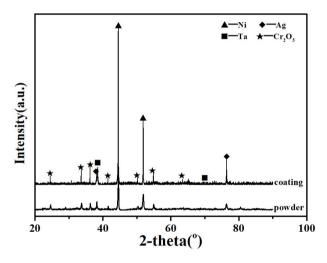
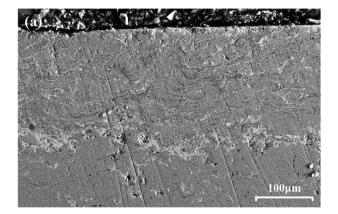


Fig 2. XRD patterns of NiAl-10Ag-5Ta-Cr<sub>2</sub>O<sub>3</sub> coating and powder



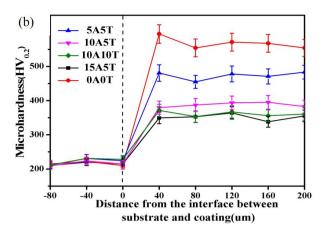


Fig. 3. (a) SEM images and (b) micro hardness of cross-section of NiAl-Ag-Ta-Cr\_2O\_3 coatings

Fig. 3(a) shows the cross section morphology of NiAl-10Ag-5Ta-Cr<sub>2</sub>O<sub>3</sub> coating, it shows a typical lamellar structure with a thickness of about 300 µm, consists of ribbon-like layers, and tightly combined each other with less pores or cracks. Benefit from the suitable deposition conditions and the reaction of Al-coated Ni power, the interface between substrate and coating show less cracks. Through the element distribution test, the bright and white areas mainly consisted of Ni and Al, and the black area mainly consisting of Cr<sub>2</sub>O<sub>3</sub> with stripy distribute. Due to the heterogeneous distribution of the compositions of deposited coatings may lead to the maldistribution of Vickers hardness values. the microhardness of section was conducted at 40 µm interval to evaluate mechanical property of composite coatings. From the results in Fig. 3(b), the microhardness of NiAl-Cr<sub>2</sub>O<sub>3</sub> coating (595 HV) is much higher than NiAl coating (180 HV) due to the reinforcement of Cr<sub>2</sub>O<sub>3</sub>. The composite coatings with containing soft metal (Ag and Ta) show a lower microhardness. And the microhardness of 15A5T coating significantly decreases to 350 HV due to the higher sliver contents (15 wt.%).

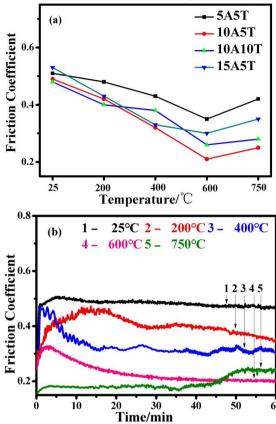
## 3.2. Tribological properties

Fig. 4(a) further gives the Cof curves of 10A5T coating at RT to 750  $^{\circ}$ C, the Cof is relatively stable as sliding distance increases, and sustain in relatively lower value.

Fig. 4(b) shows the average coefficient of friction (Cof) of NiAl-Ag-Ta-Cr<sub>2</sub>O<sub>3</sub> coatings sliding against Si<sub>3</sub>N<sub>4</sub> 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 a lower contents Ta and Ag shows the highest Cof values at all temperatures due to the insufficient lubrication phase. As the contents of Ag increases to 10 wt.%, the Cof of 10A5T coating slightly decreases at RT and 200 °C, and significant 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). And the Cof of 15A5T coating with a higher Ag is only lower than the 5A5T coating at all temperatures, this may due to the excessive lubricants lead to mechanical properties (473-351 HV) decreased. The Cof of all coatings has no much different at RT and 200 °C

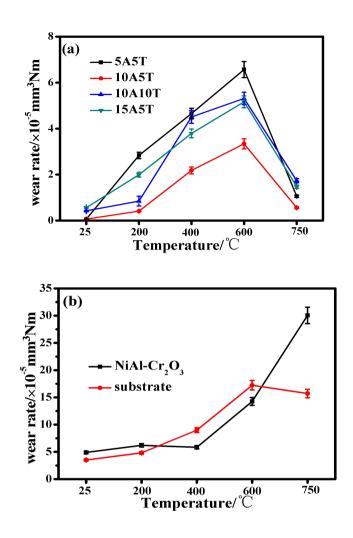


compared with each other, and the 10A5T coating shows a lower value at all temperature, especially at 600  $^{\circ}$ C (0.22).



**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

The wear rates of composite coatings, NiAl-Cr2O3 and substrate at RT to 750 °C were shown in Fig. 5(a), (b). Apparently, the wear rate of NiAl-Ag-Ta-Cr<sub>2</sub>O<sub>3</sub> coatings is much lower than the NiAl-Cr<sub>2</sub>O<sub>3</sub> at all temperatures, while it indicates 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<sub>2</sub>O<sub>3</sub> coatings show an increased wear rate, and get a maximum at 600 °C, then decline sharply at 750 °C. At RT, the 15A5T and 10A5T coating shows the lowest wear rate (10-<sup>7</sup> mm<sup>3</sup>/Nm orders of magnitude). At 200 °C, the wear rate of 10A5T and 10A10T coating (4.07×10<sup>-6</sup> mm<sup>3</sup>/Nm, 8.45×10<sup>-6</sup> mm<sup>3</sup>/N m) is lower than that of 5A5T and 15A5T coating. And the 10A5T coating has the lowest wear rate (2.18×10<sup>-5</sup> mm<sup>3</sup>/Nm) at 400 °C, this may due to the appropriate contents of lubricant and relatively high H. At 600 °C, the wear rate of NiAl-Ag-Ta-Cr<sub>2</sub>O<sub>3</sub> coatings increased, and the 10A5T coating still shows the lowest wear rate  $(3.336 \times 10^{-5} \text{ mm}^3/\text{N m})$ . Unexpectedly, the wear rates (1.057, 0.558, 1.735, 1.473×10<sup>-5</sup> mm<sup>3</sup>/Nm) sharply decreased at 750 °C, and the 10A5T coating reduces to a magnitude of 10<sup>-6</sup> mm<sup>3</sup>/Nm.

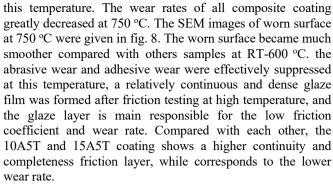


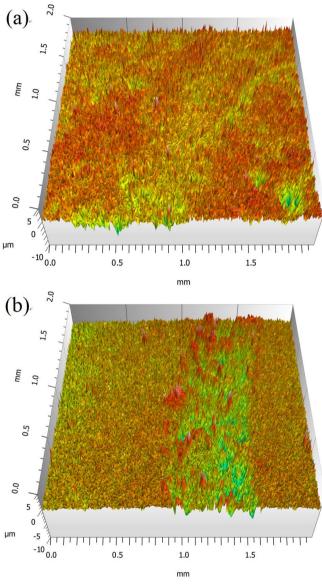
**Fig. 5.** (a) Wear rate of NiAl-Ag-Ta- $Cr_2O_3$  coatings at 25, 200, 400, 600 and 750 °C respectively; and (b) Wear rate of NiAl- $Cr_2O_3$  and substrate at 25, 200, 400, 600 and 750 °C respectively

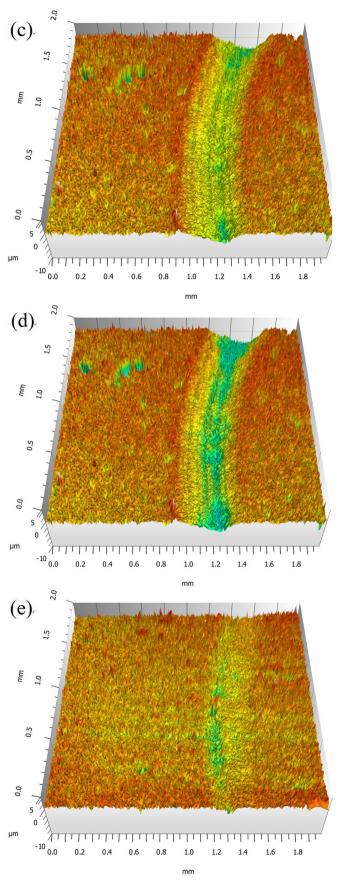
#### 3.3. Analysis of worn surface

To figure out the abrasion wear form under different temperatures, the influence of temperature on the morphologies of worn surface was discussed. Fig. 6(a)-(e) shows 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 was obtained at 600 °C, and sharply decreased at 750 °C. Besides, the worn surface was much smoother and lighter, it indicates that Ag and Ta play an important role for the wear reduction at this temperature. In addition, the worn surface SEM images of NiAl-10Ag-5Ta-Cr<sub>2</sub>O<sub>3</sub> coating at RT to 600 °C were 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. And it is more serious at 200 °C, some un-exfoliated lamellar patches can be observed due to the delamination. The worn surface was much smoother at 400 °C. As shown in fig 7(d), the worn surface shows evident adhesion wear and plastic deformation at 600 °C, while the composite coating suffered most serious abrasion at









**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  $^{\circ}$ C

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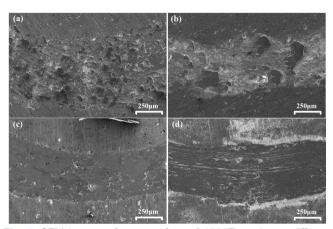
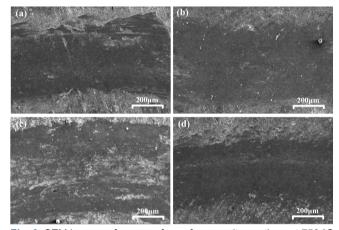


Fig. 7. SEM images of worn surface of 10A5T coating at different temperature: (a), (b), (c) and (d) respectively corresponding 25, 200, 400 and 600  $^{\circ}$ C



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

## 3.4. Discussion

To figure out the lubrication and wear resistance of NiAl-Ag-Ta-Cr<sub>2</sub>O<sub>3</sub> composite coatings, 10A5T coating was selected as the research object, its worn surface compositions under different temperatures were analyzed by EDS and Raman. Table. 3 shows the elements of worn and unworn surface at 400, 600 and 750 °C. Fig. 9 shows the corresponding raman spectrograms. In Fig 9(a), the weak characteristic peaks of AgTaO<sub>3</sub> and Ta<sub>2</sub>O<sub>5</sub> are presented in area A and B, and the much stronger characteristic peaks of Ta2O5 are existed in area C and D, and from Fig. 9(b), it indicates the worn surface mainly consist of the discontinuous oxide layer and uncovered Cr<sub>2</sub>O<sub>3</sub> and Ni. At 600 °C, a large amount of oxides formed due to enhanced oxidation reactions, the obvious characteristic peaks of AgTaO<sub>3</sub> appeared at 700 cm<sup>-1</sup> in Raman spectra, indicated that the tantalum oxides mainly exist in the form of AgTaO<sub>3</sub> and Ta<sub>2</sub>O<sub>5</sub>, while the wear rate was the highest at this temperature. As we know, in oxidation wear, the volume lost mainly comes from the wear of the oxide layer. It has been 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 dense and attached not well, the volume lost mainly comes from the oxide layer in this temperature.

From Fig. 8(b) and Fig. 9(e), a more continuous and relatively complete glaze film formed after the friction test at 750 °C, it is responsible for the increased wear resistance. The EDS results (Table 3) show that the Ag and Ta contents were higher in the worn regions compared to the unworn surfaces and other temperature regions for the glaze layer, this mainly due to the lubricant Ag with low shear stress was easy smeared on the worn surface during sliding, in addition, the Raman intensity of AgTaO3 and Ta2O5 are much stronger, as we know, AgTaO<sub>3</sub> has perovskite cubic structure, it can be produced during friction above 507 °C, the melting point of AgTaO3 is 1185 °C [15], while the friction coefficient of pure AgTaO<sub>3</sub> coating was 0.06 at 400 °C and the friction coefficient of TaN-Ag coating was 0.23 at 750 °C [16-18]. The formed frictioninduced oxidation and high temperatures oxidation (Ta<sub>2</sub>O<sub>5</sub>, Cr<sub>2</sub>O<sub>3</sub> and NiO etc.) provided wear resistance as support phase. Thus the Ag-rich glaze layers played a key role in decreasing the friction coefficient and wear rate. Above all, according to the SEM images (Fig. 7, 8 and 10), 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 property of matrix are responsible for the antifriction and wear resistance. At middle temperature (400-600 °C), the volume lost mainly comes from the oxide layer, the appropriate contents of lubricant and better mechanical properties can effectively reduce wear rate and friction coefficient for NiAl-10Ag-5Ta-Cr2O3 coating. At higher temperatures (750 °C), the tribological properties are improved by the tribo-chemical products and oxides, the relatively continuous distribution glaze layers on the worn surface was mainly consisted of AgTaO<sub>3</sub> Cr<sub>2</sub>O<sub>3</sub>, Ta<sub>2</sub>O<sub>5</sub> and Ag, which played a crucial role in improving the anti-wear and friction reducing abilities at high temperature.

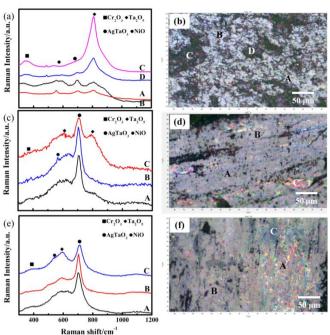
TABLE 3. Element contents of worn and unworn surface of 10A5T

coating at 400, 600 and 750 °C										
Element	400 °C	400 °C	600 °C	600 °C	750°C	750 °C				
(wt. %)	unworn	worn	unworn	worn	unworn	worn				
0	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				

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

# 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 presented at 600 °C, while the lowest wear rate in the order of  $10^{-7}$  mm<sup>3</sup>/Nm was obtained at RT, the wear resistance and lubrication of the coating mainly rely on Cr<sub>2</sub>O<sub>3</sub> and Ag at the temperature below 400 °C.

The wear rate at 750 °C was prominent ( $5.58 \times 10^{-6} \text{ mm}^3/\text{Nm}$ ), the composited glaze layers which mainly consisted of AgTaO<sub>3</sub> Cr<sub>2</sub>O<sub>3</sub>, Ta<sub>2</sub>O<sub>5</sub> and Ag are proposed to be the main reasons for reducing friction coefficient and anti-wear at 750 °C.

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