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FRICTION AND WEAR PERFORMANCE OF AN ELECTROSPARK-DEPOSITED Ta COATING ON CrNi3MoVA STEEL

TRENJE IN OBRABA PREVLEK NA OSNOVI Ta NAPRŠENIH Z ELEKTRO-ISKRILNIM POSTOPKOM NA PODLAGO IZ JEKLA VRSTE CrNi3MoV

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A Ta coating was prepared on a CrNi3MoVA steel substrate by means of electrospark deposition (ESD) and its friction and wear performance was investigated. The nanomechanical properties and friction coefficient were tested by employing a nanoindenter and a friction and wear testing machine, respectively. Moreover, the phase structure was obtained using X-ray diffraction (XRD), and the morphology and composition were analysed before and after friction utilizing scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS). The results showed that the gradient Ta coating with nanocrystals is composed of α-Ta and Fe-Ta intermetallics, and Ta_2O_5 and α-Fe phases. The hardness of the Ta coating increased by 115 % compared to that of the CrNi3MoVA steel and its elasticity was reduced by 21 %. The friction coefficient of the Ta coating was reduced by 81 % compared to that of the CrNi3MoVA steel and its weight loss was reduced by 89 %. The wear mechanism of the Ta coating can be characterized as slight adherence, whilst that of the CrNi3MoVA steel includes severe adherence and fatigue.

Keywords: Ta coating, electrospark deposition, nanomechanical properties, friction and wear

Avtorji so pripravili prevleke na osnovi tantala (Ta) na jekleni podlagi vrste CrNi3MoV z elektro-iskrilnim postopkom (ESD, angl.: electrospark deposition) in nato ugotavljali njihove tribološke lastnosti oziroma trenje in obrabo. Nano-mehanske lastnosti in koeficient trenja izdelanih prevlek so določili z nano indenterjem in recipročno napravo za določevanje trenja in obrabe (MFT-5000, angl.: reciprocating friction and wear testing machine). Fazno strukturo, morfologijo in sestavo prevlek pred in po obrabi so določili s pomočjo rentgenske difrakcije (XRD, angl.: X-ray diffraction), vrstične elektronske mikroskopije (SEM, angl.: scanning electron microscopy) in energijske rentgenske disperzijske spektroskopije (EDS, angl.: energy dispersive X-ray spectroscopy). V tem članku opisani rezultati raziskav so pokazali, da so izdelane gradientne prevleke na osnovi Ta sestavljene iz nanokristalov α-Ta, intermetalne spojine na osnovi Fe in Ta, faze Ta_2O_5 in faz α-Fe. Trdota izdelanih prevlek je bila večja za 115 % in nižja za 21 % od jeklene podlage CrNi3MoV na katero so bile le-te napršene. Koeficient trenja se je zmanjšal za 81 % in izgube zaradi obrabe so se zmanjšale za 89 %. Podlage na osnovi Ta so kazale rahlo sprijemanje z jekleno kroglico recipročne naprave za določevanje trenja in obrabe medtem, ko je bila jeklena podlaga iz CrNi3MoVA brez prevleke izpostavljena močni obrabi zaradi lepljenja in obrabi zaradi dinamičnega utrujanja materiala.

Ključne besede: prevleke na osnovi tantala, nanašanje prevlek z elektro-iskrilnim postopkom, nano-mehanske lastnosti, trenje in obraba

1 INTRODUCTION

The wear of a sliding or rolling surface can cause premature failure of a key component, and thus improper working of the whole equipment. According to some estimates, losses can amount to about 4 % of the gross national product in the United States.[1] Therefore, the significance of friction reduction and wear control should never be underestimated for economic reasons and long-term equipment operation reliability. An improved application of tribological principles can save about 1 % of a gross national product in industrialized countries, and the savings are expected to be 50 times the research expenditure.

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As wear always takes place on the interaction interface of two or more components, coating technologies, such as laser cladding,[2] magnetron sputtering,[3] electrospark deposition (ESD),[4,5] etc., have been most widely applied to improve the surface tribological property. Among all these coating technologies, ESD has many noticeable advantages for depositing wear resistance coatings on a metal substrate in contrast to the other coating technologies. In an ESD process, the conductible electrode (anode) and substrate (cathode) are instantaneously pulsed-tripped and discharged, and then physical and chemical reactions take place in a melting micropool on the surface of the substrate. The electrode moves back and forth, and numerous melting micro-pools link and overlap to form metallurgically bonded coatings.[6] ESD, a simple, cost-effective and environment-friendly technique, can prevent distortion or metallurgical changes of the substrate due to a low heat input, so it has been widely applied in the fields of machinery, chemical engineering, military, aviation, etc.

Recently, the ESD technology and its application have attracted intense interest of researchers due to good properties of ESD coatings, especially their outstanding wear resistance created on a steel substrate. Yang et al.[7] prepared a NiCrAlY coating on a CrNi3MoVA substrate by means of ESD, showing that a NiCrAlY coating on the CrNi3MoVA steel allows antifriction and wear resistance mainly due to its high hardness and a well adherent, thin surface oxide layer formed during high-speed friction. In another investigation by the same authors,[8] a tribological comparison between an ESD W-Ni-Fe-Co coating and electroplating hard chromium coating was carried out; the results indicated that the former exhibited better friction and wear performance than the latter. Moreover, ESD is an environment-friendly technique in contrast to electroplating with a harmful hexavalent chromium solution.

α-Ta exhibits good ductility, high melting temperature (2996 °C), high hardness (8–12 GPa) and excellent corrosion resistance, so its coating is a desirable candidate to replace the electroplating chromium coating, prepared with the magnetron sputtering technology. It has been a research hotspot with the aim to protect a gun bore from wear and erosion since the early 1970s. However, a magnetron sputtering Ta coating always includes a part of the β-Ta phase that is very brittle and will degrade the mechanical properties of the coating[9]. Moreover, it is well known that the coatings prepared with magnetron sputtering exhibit poor bonding with the substrate, which is of key importance for an engineering application. In contrast to magnetron sputtering coatings, ESD coatings have strong bonding with the substrate and do not need a complex vacuum system. Thus, it is desirable to prepare a Ta coating by means of the ESD technology when dealing with a gun bore to improve its service life. To the best of our knowledge, there is still no relevant research report about a Ta coating on a gun steel substrate prepared with ESD. Hence, in this work, the tribological properties of an ESD Ta coating on a CrNi3MoVA steel substrate is investigated.

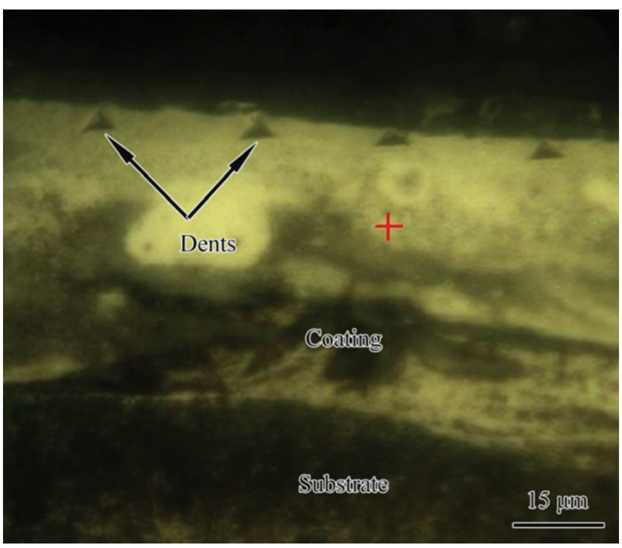

Figure 1: Indentation positions of the hardness and elasticity modulus test on the cross-section of Ta coating

2 EXPERIMENTAL PART

CrNi3MoVA steel was selected as the substrate sample material and its chemical composition is shown in **Table 1**. A bar of the CrNi3MoVA steel was processed into rectangle-shaped samples with dimensions of (15 × 10 × 4) mm, and a Ta bar with a purity of 99.999 % was processed into a cylinder-shaped electrode with dimensions of Φ4 × 50 mm by employing wire electrical discharge machining (WEDM). The samples and electrode were ground using SiC abrasive paper to 800-grit finish and ultrasonically cleaned in an ethanol and acetone mixture. The Ta coating was prepared utilizing a DJ-2000 adjustable power metal surface repairing machine, and the preliminary optimized processing parameters were set as shown in **Table 2**. For the convenience of the discussion, thereafter a sample coated with Ta is denoted the Ta coating.

Table 1: Chemical composition of CrNi3MoVA steel[10]

C	Mn	Si	Cr	Ni	Mo	V	S	P
0.40	0.41	0.25	1.28	3.14	0.37	0.20	0.001	0.012

Table 2: Preliminary optimized processing parameters of ESD

Power /W	Voltage /V	Ar gas flow /L·min^{-1}	Electrode rotating rate /r·min^{-1}	Deposition time unit area /min·cm^{-1}
1200	60	15	3000	1

The hardness and elasticity modulus were tested with a G200 nanoindention instrument with a Berkovich indenter and calculated with the Oliver-Pharr method through loading curves. Nanoindentation is a technique that tests the mechanical properties of a material on the sub-microscale. **Figure 1** presents the indentation positions of the hardness and elasticity modulus test on the cross-section of the as-deposited Ta coating. As the surface of the as-deposited Ta coating is not even, its hardness and elasticity modulus were tested on its polished cross-section near the coating surface (as shown in **Figure 1**), and the obtained values were the average values of eight parallel test points. The friction and wear were tested using an MFT-5000 reciprocating friction and wear testing machine. The test conditions were as follows: the grinding head was a quenched GCr15 steel ball with a diameter of 9.5 mm, the reciprocating distance was 6 mm, the reciprocating speed was 5 mm per second, the load was 20 N and the total friction time was

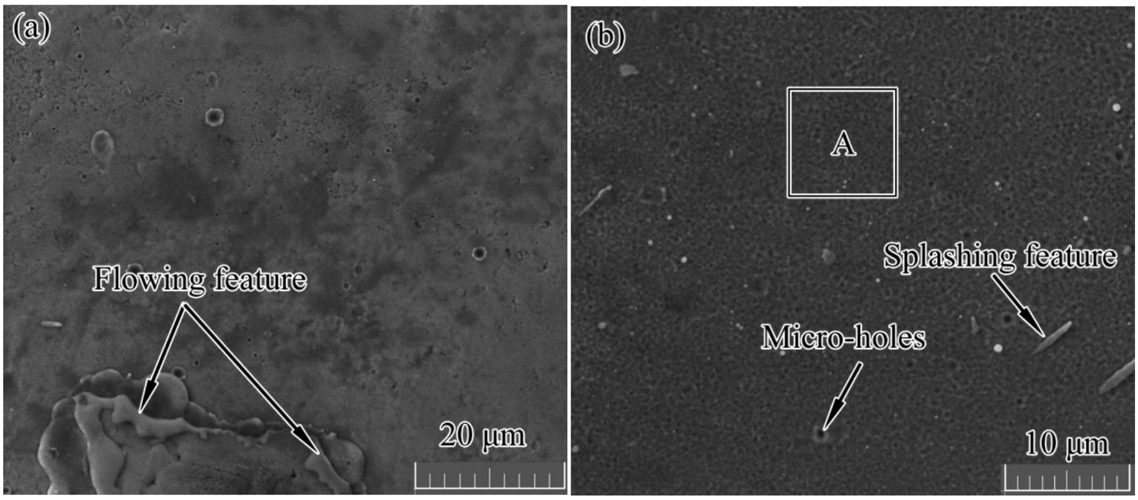

Figure 2: a) Surface morphology at low magnification and b) high magnification of the as-deposited Ta coating

30 min. The mass of the worn samples was weighted with an electronic balance (Sartorius BP211D) with a sensitivity of 10^{-5} g after being ultrasonically cleaned within the ethanol and acetone mixture for 60 min.

Surface morphologies were obtained with scanning electron microscopy (SEM, TESCAN MAIA3, Tescan Co., Brno, Czech Republic), while an energy-dispersive spectrometer (EDS, X-Max, Oxford Instruments Co., Oxford, UK) was used to analyse the chemical composition of the selected area. The phase constitution of the coating was identified with X-ray diffraction (XRD, X' Pert PRO, PANalytical Co., Almelo, Holland), a technology for analysing a crystal structure based on the diffraction effect of X-rays in a crystal.

3 RESULTS

3.1 As-deposited Ta-coating microstructure

Figure 2 shows the surface morphology under low magnification (a) and high magnification (b) of the as-deposited Ta coating. As shown in **Figure 2a**, the surface of the as-deposited Ta coating is smooth, except that there is a molten-material flowing edge feature on it. On the high-magnification surface (**Figure 2b**), a few micro-holes and a splashing feature caused by molten material splashing can be carefully observed. EDS results indicate that the composition of region A includes 48.78 at.% Ta, 47.65 at.% Fe, 1.79 at.% Ni and 1.78 at.% O, suggesting that although it is prepared under argon, the as-deposited Ta coating is still oxidized, including a small quantity of oxygen.

Figure 3 shows the cross-section (a) and magnification of region B (b) of the as-deposited Ta coating. In order to distinguish between the as-deposited Ta coating and the substrate, the cross-section of the as-deposited Ta coating was etched using 4 % Nital (by volume). As shown in **Figure 3a**, the coating is dense and has excellent bonding with the substrate. **Table 3** shows the EDS results for points 1–8 on the cross-section of the as-deposited Ta coating from **Figure 3a**. As shown in **Table 3**,

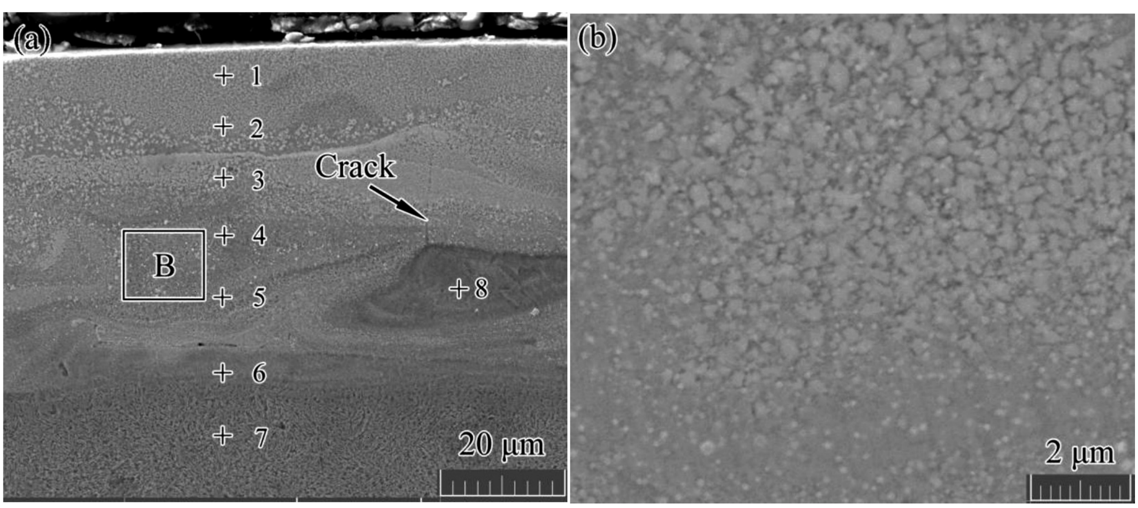

Figure 3: a) Cross-section and b) magnification of region B of the as-deposited Ta coating

from the top of the coating to the substrate, the content changes of Ta and Fe are opposite. Moreover, at the interface of the coating and the substrate, the contents change gradually, indicating that the as-deposited Ta coating is a gradient one. The EDS image of point 8 shows that the dark region in the cross-section of the coating contains less Ta, which is in contrast to the horizontal position where a crack can be observed. The magnification of region B (**Figure 3b**) shows that the as-deposited Ta coating consists of fine grains with a microcrystalline and even nanocrystalline size.

Table 3: EDS results for points 1–8 on the cross-section of the as-deposited Ta coating from **Figure 3a**

Element	Ta	Fe	Ni
Point 1	40.63	58.13	1.24
Point 2	39.78	58.05	2.17
Point 3	33.33	64.95	1.72
Point 4	26.15	71.75	2.10
Point 5	22.38	75.72	1.90
Point 6	14.13	83.40	2.47
Point 7	0	96.71	3.29
Point 8	4.86	92.95	2.69

Figure 4 shows the XRD pattern of the as-deposited Ta coating. Combined with the EDS results, it indicates that the as-deposited Ta coating consists of α-Ta, Fe-Ta intermetallics, Ta_2O_5 and α-Fe.

3.2 Nanomechanical properties of Ta coating and CrNi3MoVA steel

Table 4 shows the nanomechanical properties of the as-deposited Ta coating and CrNi3MoVA steel. We can see that the hardness of the as-deposited Ta coating increases by 115 % compared to that of CrNi3MoVA steel and its elasticity decreases by 21 %, enhancing an increase in H/E and H^3/E^2, two other important parameters relating to the tribological property that are discussed later.

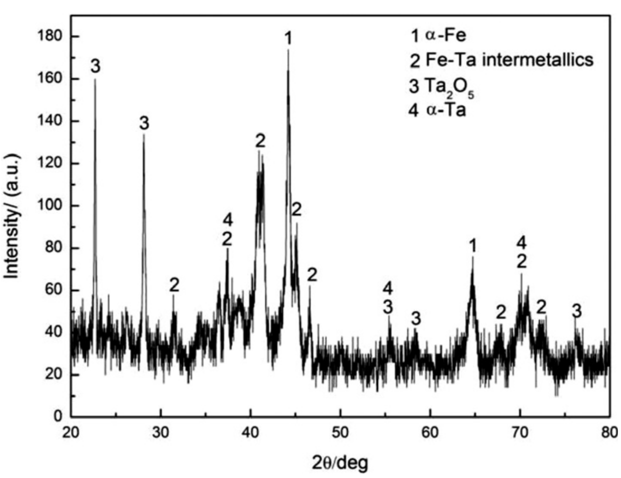

Figure 4: XRD pattern of the as-deposited Ta coating

Table 4: Nanomechanical properties of the as-deposited Ta coating and CrNi3MoVA steel

Samples	H (GPa)	E (GPa)	H/E	H^3/E^2
Ta coating	10.04	207.3	0.048	0.0236
CrNi3MoVA steel	4.68	262.8	0.018	0.0015

3.3 Friction coefficient and wear mass of Ta coating and CrNi3MoVA steel

Figure 5 shows the friction coefficient and wear mass of the Ta coating and the CrNi3MoVA steel. We can see that the friction coefficient of the Ta coating is smooth and steady, and its value is less than 0.14; the friction coefficient of the CrNi3MoVA steel has a running-in stage over about 350 s, and then it attains a steady friction and wear stage with a value of 0.73–0.75. In contrast to the CrNi3MoVA steel, the friction coefficient of the Ta coating decreases by 81 %. The weight loss of the Ta coating is 1.36 mg, while that of the CrNi3MoVA steel is 12.17 mg, and the weight loss of the Ta coating decreases by 89 % compared to that of the CrNi3MoVA steel (**Figure 5b**). Therefore, the Ta coating enhances the friction and wear performance of the CrNi3MoVA steel substrate.

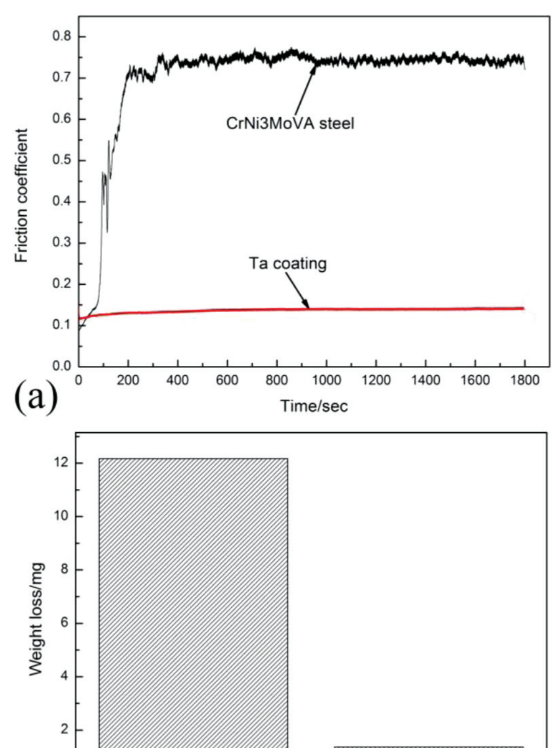

Figure 5: Friction coefficient and wear mass of the Ta coating and CrNi3MoVA steel

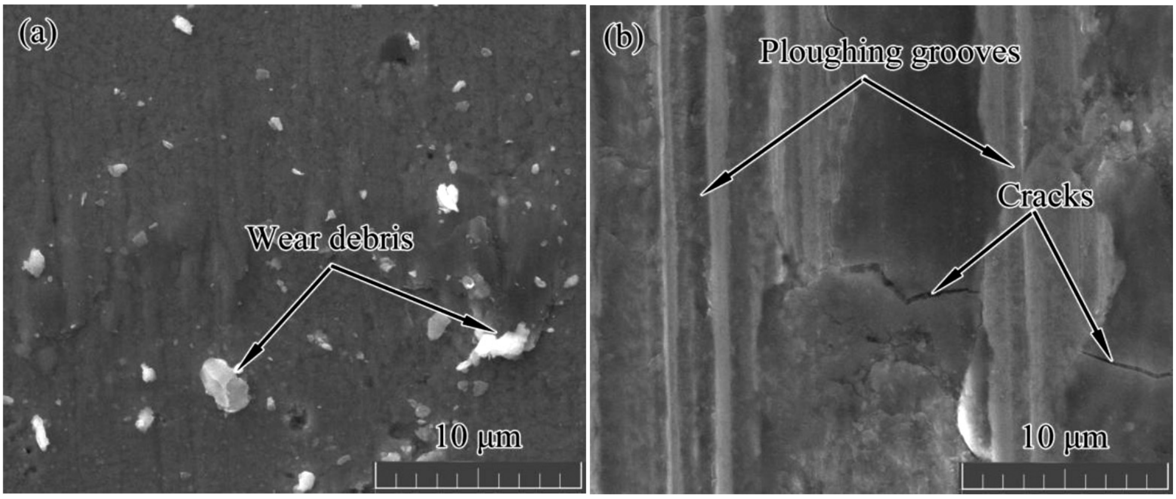

Figure 6: Worn surface morphologies of the Ta coating and CrNi3MoVA steel

3.4 Wear mechanism of Ta coating and CrNi3MoVA steel

Figure 6 shows the worn surface morphologies of the Ta coating and CrNi3MoVA steel. As shown in **Figure 6a**, in contrast to the CrNi3MoVA steel (**Figure 6b**), the micro-holes and splashing feature disappear and there are a few sparkly bits of wear debris with a size of less than two microns distributed on the smooth surface of the worn Ta coating. Therefore, the wear mechanism of the Ta coating can be characterized as slight adherence. Compared with the worn surface of the Ta coating (**Figure 6a**), there are ploughing grooves on the worn surface of the CrNi3MoVA steel, indicating that severe plastic deformation takes place under the action of a rubbing load. In addition, cracks can also be observed on the worn surface of the CrNi3MoVA steel, which suggests that the accumulated stress in the rubbing zone is beyond the fracture strength of the CrNi3MoVA steel. Thus, the mechanism of the CrNi3MoVA steel includes severe adherence and fatigue.

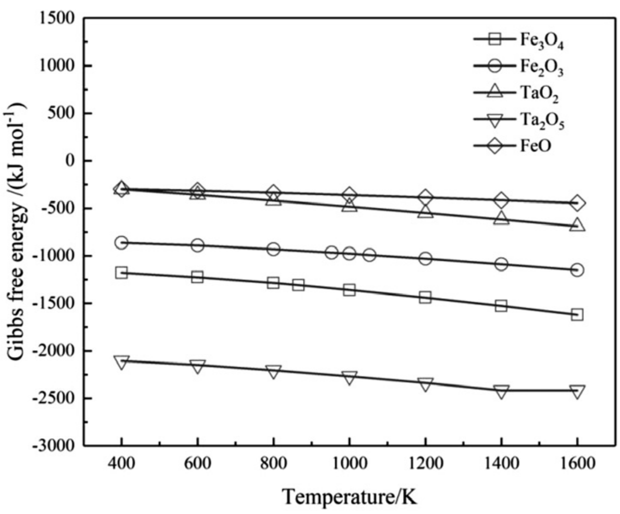

Figure 7: Gibbs free energy change in the oxides of Ta and Fe at various temperatures

4 DISCUSSION

4.1 Forming of Ta coating

In an ESD process, argon has two effects, i.e., it can facilitate a plasma with low thermal conductivity and form a spraying feature (**Figure 6b**) for the melt on the surface of an as-deposited coating, thus producing a smoother and more uniform surface morphology.[11] It can protect the coating from severe oxidation that can reduce its thickness and properties. However, recent investigations have shown that selective oxidation in an ESD process may have a beneficial influence on the properties of a coating. Yang et al.[12] prepared a Ni-MoS_2 coating on a CrNi3MoVA steel substrate using the ESD technique, showing that an amount of MoO_2 formed in-situ due to a reaction with O_2 can act as an oxide dispersion strengthened (ODS) phase in the coating. Guo et al.[13] also found that oxidation took place in argon in the ESD process when they prepared AlCoCrFeNi high-entropy alloy coatings, and they inferred that the oxide was very likely to be Al_2O_3 in light of the thermodynamics calculation. **Figure 7** shows the Gibbs free energy change in the oxides of Ta and Fe at various temperatures.[14] We can see that the Gibbs free energy of Ta_2O_5 is far smaller than that of the other oxides, so Ta_2O_5 is prone to form during an ESD process, which is consistent with our XRD patterns (**Figure 4**).

According to the Fe-Ta binary diagram[15] (**Figure 8**), solid solubility between Fe and Ta is low, so they are prone to form Fe-Ta intermetallics (**Figure 4**), which limit the thickness of an ESD Ta coating. It is well known that Fe-Ta intermetallics have high hardness and strength, and they can be the strengthened phases in the Ta coating. In an ESD process, the melting materials of the electrode and substrate produce micro-pools on the surface of the substrate in a contact discharge, and with

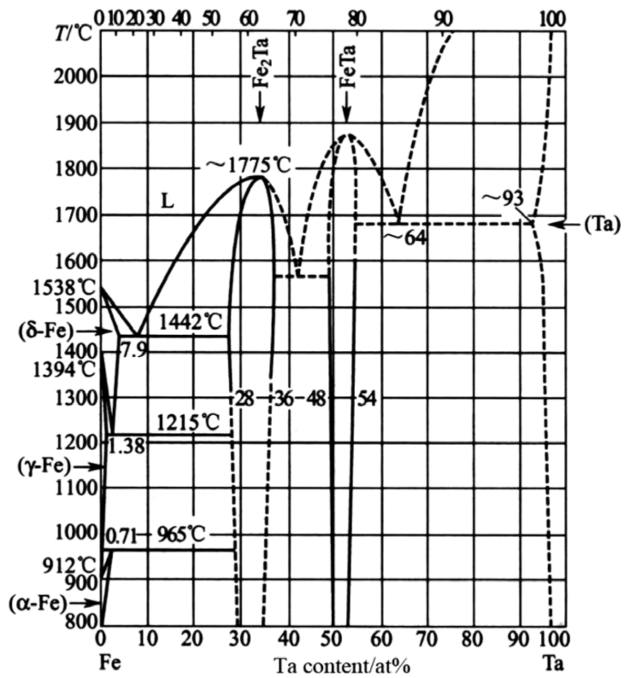

Figure 8: Fe-Ta binary diagram

the electrode moving forth and back, the micro-pools merge, forming the first layer after the electrode have moved along the whole surface of the substrate. Then the electrode moves to the previously formed layer to form the second layer and so on. Therefore, the content of Ta gradually increases from the interface to the coating surface, forming a gradient coating (**Figure 3a**). This gradation increases the compatibility of the coating and substrate properties during the rapid cooling formation of the coating. However, due to the manual operation of ESD, nonuniformity of the coating is inevitable, i.e., the dark region in the cross-section of the Ta coating (**Figure 3a**). The dark region destroys the gradation of the coating, producing a crack during the rapid cooling formation of the coating.

4.2 Wear resistance of Ta coating

Nanoindentation can provide significant parameters to predict tribological properties of samples.[16–18] The value of H/E relates to wear resistance and it determines the elasticity limit of a rubbing contact surface. With an increase in the H/E value, the quantity of asperity beyond the elasticity limit is reduced on the rubbing contact surface under the action of stress, increasing the wear resistance for antifriction. According to **Table 4**, the H/E value of the Ta coating increases about 1.6 times compared to that of the CrNi3MoVA steel, indicating that the Ta coating has better wear resistance than the CrNi3MoVA steel. Another parameter, the H^3/E^2 value, also relates to wear characteristics of samples, showing the capability to resist plastic deformation under the action of a contact load, i.e., the yield pressure. According to **Table 4**, the H^3/E^2 value of the Ta coating increases about 14.7 times compared to that of the CrNi3MoVA steel, which also suggests that the Ta coating has better wear resistance than the CrNi3MoVA steel.

The outstanding wear resistance of the Ta coating is substantially attributed to its microstructure and main phases. The Fe-Ta intermetallics and Ta_2O_5 with high hardness and strength can be dispersed strengthened phases in the matrix of α-Ta and α-Fe with excellent toughness. Resulting from the nanocomposite strengthening effect, the Ta coating has a much higher hardness than the CrNi3MoVA substrate, and in terms of Archard's law,[19] the sliding wear resistance is proportional to the alloy hardness. Furthermore, the microstructure of the Ta coating is dense, without porosity (**Figure 3a**) and this feature prevents the abrasive particles from micro-cutting the coating surface.

5 CONCLUSIONS

A gradient Ta coating with nanocrystals was prepared by means of the ESD technique and it is composed of α-Ta, Fe-Ta intermetallics, Ta_2O_5 and α-Fe phases. Nanoindentation results showed that the hardness of the Ta coating increased by 115 % compared to that of the CrNi3MoVA steel and its elasticity decreased by 21 %. Friction and wear tests revealed that the friction coefficient of the Ta coating decreased by 81 % compared to that of the CrNi3MoVA steel and its weight loss decreased by 89 %. The wear mechanism of the Ta coating can be characterized as slight adherence, whilst the mechanism of the CrNi3MoVA steel includes severe adherence and fatigue. These results indicate that the ESD Ta coating has an excellent antifriction and wear resistance and can greatly improve the tribological performance of key components made of steel.

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