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Fabrication and High Temperature Friction Behavior and Oxidation Resistance of Ni-Co-ZrO_2 Composite Coating

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Ni-Co alloy and ZrO_2 micron particles were codeposited on 45 carbon steel by electrodeposition. The composition and microstructure of the composite coating were characterized. The high temperature tribological properties were carried out by a pin-on-disk tribo-tester. Additionally, the oxidation resistance was evaluated via high temperature circulating oxidation test. The results indicated that the deposited composite coating showed dispersed ZrO_2 particles and continuous Ni-Co matrix, and there were no obvious pores, cracks and other defects at the interface between the composite coating and the substrate. The embedded ZrO_2 particles changed the friction mechanism from adhesive wear to abrasive wear, the wear loss rate and friction coefficient of Ni-Co-ZrO_2 composite coating were lower in comparison with that of Ni-Co alloy coating and carbon steel substrate. In addition, the embedded ZrO_2 particles exerted a reactive-phase effect on the growth of nickel oxide and cobalt oxide, and effectively reduced the oxidation rate of the substrate at high temperature. Therefore, the Ni-Co-ZrO_2 composite coating presents better oxidation resistance, when compared with Ni-Co coating.

Keywords: *Electrodeposition, Ni-Co-ZrO_2 composite coatings, High temperature tribological behavior, Oxidation resistance*

1. Introduction

In modern industries, carbon steels are widely used as a structural material for a variety of engineering applications. However, carbon steels are easy to suffer an attack in aggressive solutions and atmospheres because of its high corrosion and oxidation rate [1]. Moreover, many carbon steel mechanical products and parts should be able to work steadily for long term, especially under high temperature, high-pressure and high-speed conditions. For example, in hot forging processing, works failed due to high temperature wear is a serious problem, and 70-80% of dies in hot environments are damaged by wear. The prolonged life and reduced cost of die is achieved by the use of the anti-wear materials in high temperature environments [2]. Therefore, in order to improve the wear, corrosion and high temperature oxidation resistance of carbon steels, surface strengthening techniques are still needed to increase the service life and reliability and to improve the performance and quality of mechanical equipment. Fine particles reinforced metal matrix composite coatings are made up of matrix metal and evenly dispersed second-phase particles such as ZrO_2 [3], B_4C [4], Si_3N_4 [5], WC [6], Al_2O_3 [3,5,7], CeO_2 [8], SiC [5,9,10], possessing excellent comprehensive performance. ZrO_2 is commonly used as the second ceramic phase due to its high hardness, low thermal conductivity and high temperature oxidation resistance. At the same time, Ni-Co alloys are important as they possess high temperature wear and corrosion resistance because it is hardened by the addition of cobalt (Co) into nickel (Ni) in a form of solid solution which does not embrittle during the heat treatment [11-12]. For Ni-Co alloys possess excellent mechanical and chemical properties, incorporating ZrO_2 particles into Ni-Co matrix achieved by electrodeposition presents particular chemical and physical properties, leading to a new class of composite coating [7-9]. Currently, most research works focus on improving the hardness, corrosion and wear resistance, and electrocatalytic activity of Ni-Co alloy matrix composite coatings [7-9,13]. However, the researches on high temperature tribological behavior and the high temperature oxidation resistance of Ni-Co-ZrO2 composite coating have been less reported yet. In this work, submicron ZrO2 particles reinforced Ni-Co alloy composite coating was prepared by electrodeposition and its high temperature tribological behavior and its high temperature oxidation resistance were investigated. In the meantime, the phase structure and surface morphologies were also analyzed. For comparison, the Ni-Co alloy coating was also prepared.

2. Experimental details

The electrodeposition was carried out using a ZD-A direct current power supply. Two nickel samples with the size of 70×20×5 mm3 were used as the anode, and 45 steel sample with the size of 12×12×5 mm3 was used as the cathode. The average particle size was about 0.58 µm. ZrO2 particles were subjected to hydrochloric acid with a concentration of 37.5% for 6 h to remove metal impurities that may exist. The particles were flushed with distilled water till neutral and dried for later use. Prior to the co-deposition,

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the agglomerated particles were dispersed by suspending the ZrO2 particles in the electrolyte and subjecting to ultrasonic in the bath for 4 h. The bath was stirred by a magnetic stirrer and maintained at the required temperature. The duration time for plating was maintained so as to obtain a thickness of 65 µm. Ni-Co alloy coating and Ni-Co-ZrO2 composite coating were prepared under the same conditions of deposition (seen from Table 1).

High temperature friction and wear test was performed under dry sliding condition at 873 K for 15 min by a rotational pin-on-disk tribo-tester (HT-1000, Lanzhou Zhongkai LTD. China). Si3N4 ball (hardness > HV1300) was used as the counter-body. The tests were conducted on a track radius of 5 mm under a load of 10 N and a sliding speed of 0.293 m/s for 15 min. The friction coefficients and sliding time were recorded automatically during the test. Each of friction pairs was cleaned by ultrasonic washing in acetone before and after each test. An electrical balance was used to weigh the samples before and after each wear test, so as to calculate the weight loss of the coatings.

High temperature oxidation tests were carried out in a muffle furnace in static air. The temperature of the furnace was maintained at a set value by an automatic controller with precision of ±2 K. The oxidation experiments were carried out at 573 K, 873 K and 1173 K for 2 h, respectively, and at 873 K for different exposure time [14-16]. After oxidation, the samples were withdrew from the furnace and cooled in air without air flow, and then weighed the samples by using an electronic balance with an accuracy of 0.01 mg.

Table 1. Composition of plating solution and experiment conditions

$NiSO_4 \cdot 6H_2O$	210 g/L
$CoSO_4 \cdot 7H_2O$	95 g/L
H_3BO_4	20 g/L
Sulfourea	10 mg/L
ZrO_2 (0.58 µm)	60 g/L
Parameters for equipment setup	
Current density	8 A/dm^2
Magnetic stirring rate	250 rpm
Temperature	328 K
pH	2.5~3.5
Plating duration	80 min

The surface and cross-section micrographs of Ni-Co coatings and Ni-Co-ZrO_2 composite coatings before and after high temperature oxidation test, as well as the surface morphologies of the coatings after wear test were observed by Scanning Electron Microscopy (SEM, JSM-6390A, Japan)) and the chemical composition of the Ni-Co and Ni-Co-ZrO_2 composite coatings before and after high temperature oxidation test were probed by Energy Dispersive Spectroscopy (EDS) attached to the above-mentioned SEM. Phase analysis of the Ni-Co-ZrO_2 composite coating after high temperature friction test was identified by X-ray diffraction (XRD) (XRD-6000, Japan) with a CuKα radiation.

3. Results and discussion

3.1 Morphology observation and EDS analysis

Fig. 1(a) shows the surface SEM micrographs of the Ni-Co coating. It is clearly that Ni-Co coating presents typical crystal morphology with average uniform particle size of about 4 µm. The larger crystal particles size makes the coating surface coarse relatively. Fig. 1(b) shows the surface SEM micrographs of the Ni-Co-ZrO_2 composite coating prepared on 45 steel substrate. It can be seen that the obtained composite coating possesses a mat-gray smooth metallic surface with fine and compact white spots which are visible to naked eye. And in Fig. 1(d), we can see that each white ZrO2 particle size is less than 1 um, it means the distribution of ZrO2 particles is favorable. Fig. 2(a) presents the cross-section micrograph of Ni-Co composite coating, showing continuous and good combinations between the coating and the substrate, and there are no obvious defects between the composite coating and the substrate. And Fig. 2(b) shows the cross-section micrograph of Ni-Co-ZrO2 composite coating, which presents dispersed ZrO2 particles and continuous Ni-Co matrix, and there are no pores and cracks and other defects at the interface.

Fig. 3 (a) shows the EDS analysis which shows that the composition of coating is Ni-Co alloy and there are no impurities in the coating. Fig. 3 (b) illustrates the EDS analysis data, which strongly suggest that the composition of coating are nickel cobalt (Ni-Co) alloy and ZrO2 particles, and it also reveals much higher content of cobalt (67.69wt%) than that of nickel (29.68wt%) in the composite coating. Co-rich

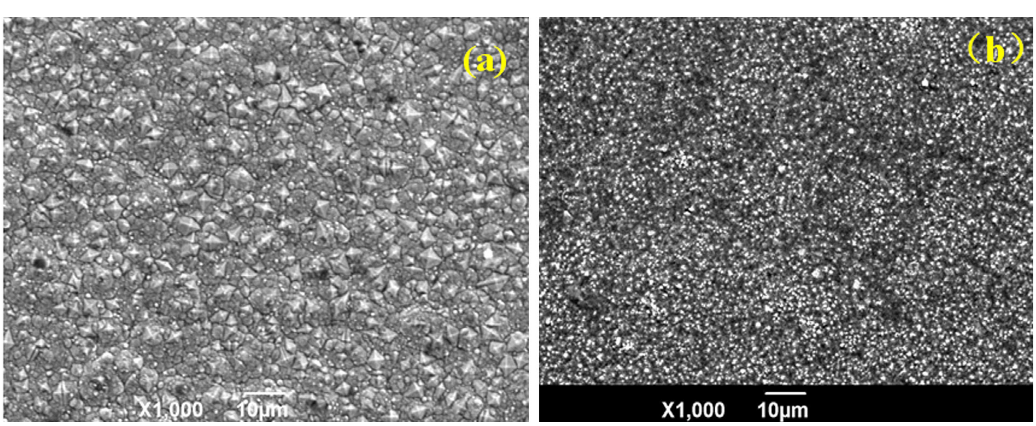


Fig. 1 SEM micrographs of surface: (a) Ni-Co coating (b) Ni-Co-ZrO2 composite coating

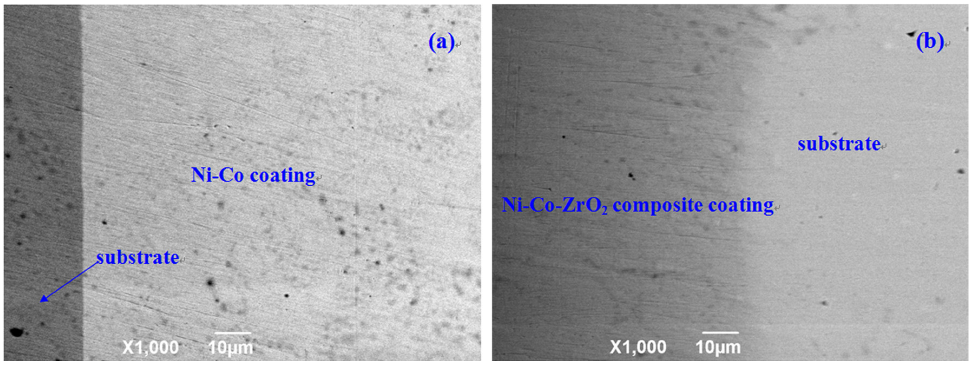
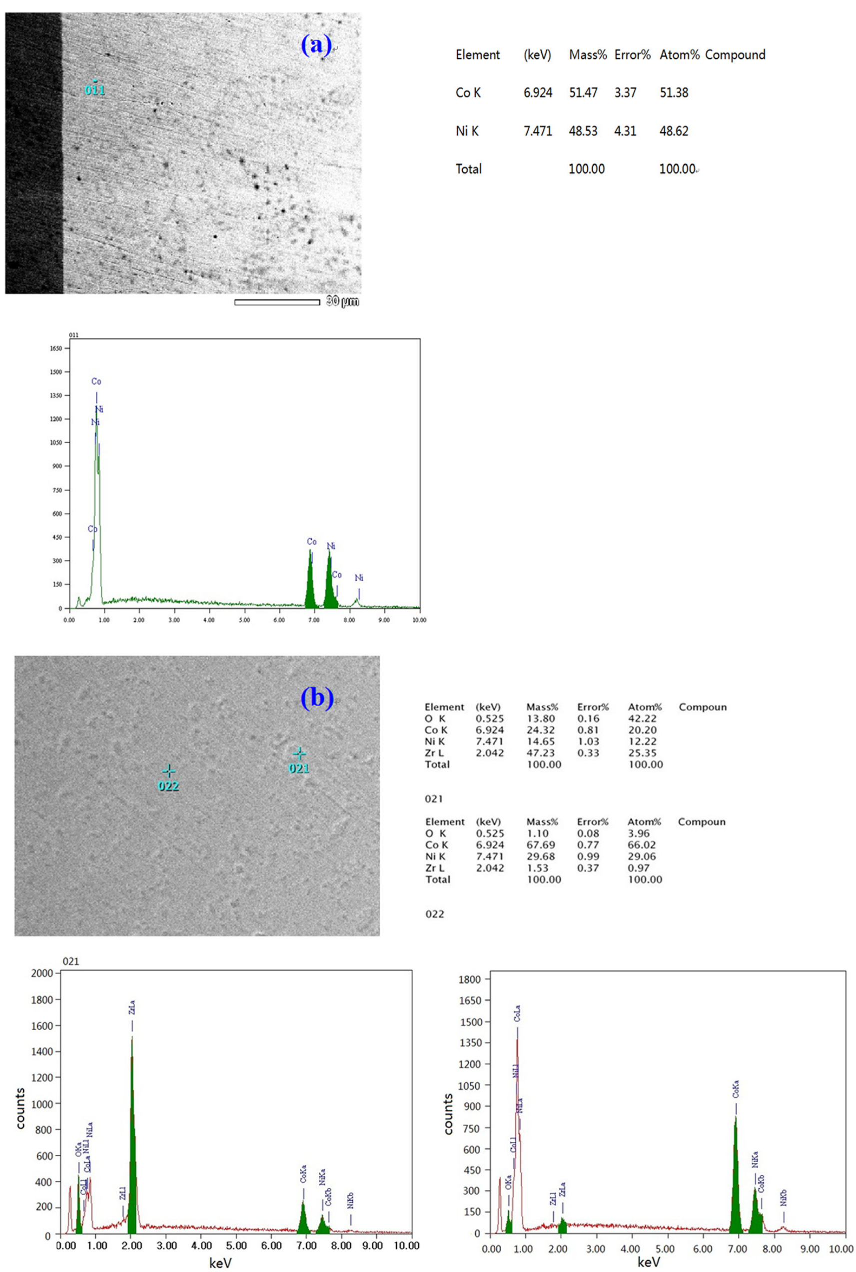


Fig. 2 SEM micrographs of the cross-section: (a) Ni-Co coating (b) Ni-Co-ZrO2 composite coating.



Element	(keV)	Mass%	Error%	Atom%	Compound
Co K	6.924	51.47	3.37	51.38	
Ni K	7.471	48.53	4.31	48.62	
Total		100.00		100.00	

Element	(keV)	Mass%	Error%	Atom%	Compoun
O K	0.525	13.80	0.16	42.22	
Co K	6.924	24.32	0.81	20.20	
Ni K	7.471	14.65	1.03	12.22	
Zr L	2.042	47.23	0.33	25.35	
Total		100.00		100.00	

021

Element	(keV)	Mass%	Error%	Atom%	Compoun
O K	0.525	1.10	0.08	3.96	
Co K	6.924	67.69	0.77	66.02	
Ni K	7.471	29.68	0.99	29.06	
Zr L	2.042	1.53	0.37	0.97	
Total		100.00		100.00	

022

Fig. 3 EDS analysis of (a) Ni-Co coating (b) Ni-Co-ZrO2 composite coating.

Ni-Co alloy possesses better thermostability compared with Ni-rich Ni-Co alloy [17], so the coating was designed with higher Co content than Ni in content. Fig. 3(b) also shows that the embedded ZrO2 particles with irregular shape homogeneously disperse within the Ni-Co matrix.

3.2 High temperature friction behavior of Ni-Co-ZrO_2 composite coating

Fig. 4 illustrates the variations of wear weight loss for 45 steel substrates, Ni-Co alloy coating and Ni-Co-ZrO_2 composite coating. The wear rate decreases from 25.1×10^{-3} mg/m for 45 steel down to 22.3×10^{-3} mg/m for Ni-Co coating and further down to 14.8×10^{-3} mg/m for Ni-Co-ZrO_2 composite coating. Fig. 4 demonstrates that Ni-Co-ZrO_2 composite coating possesses the best wear resistance. The decrease of the wear rate of Ni-Co-ZrO_2 composite coating, as compared with Ni-Co coating, is rationally understood because of the plastic deformation of the matrix material under the load by way of the ZrO_2 particles dispersion strengthening.

Fig. 5 shows the relationships between friction coefficient and the sliding time for 45 steel, Ni-Co alloy coating and Ni-Co-ZrO_2 composite coating at 873 K. It shows that the friction coefficient of Ni-Co-ZrO_2 composite coatings is lower than that of Ni-Co coating and much lower than that of 45 steel, which agrees well with the results shown in Fig. 4.

Fig. 6 presents the XRD pattern of Ni-Co-ZrO_2 composite coatings after high temperature friction at 873 K for 15 min. The peaks of NiO and CoO phases which could further prevent inward oxygen diffusion, appear on the XRD pattern, and there are no ferric (Fe) oxides, confirmed by Fig. 3, where no Fe was probed, It shows that the carbon steel substrate is well protected by the composite coating.

Fig. 7 shows the morphologies of the wear traces surface of 45 steel with and without coatings. Fig. 7(a-b) presents photographs of a typical worn surface of 45 carbon steel at 873 K, Fig. 7(a) shows that there is a wide and dark furrow along the sliding direction. From Fig. 7(b), it can be seen that the entire surface of 45 steel after oxidation is dark, the oxidation is heavy, and it further identifies that the darker areas in Fig. 7(a) are indeed a deep groove. The groove means that the wear loss will be high. The result as just mentioned could be attributed to the brittle oxide layer, which cannot effectively prevent the substrate from sliding cut from the micro-contact surface of counter-part [16].

Fig. 7(c-d) presents the wear trace of Ni-Co coating. Arrows in Fig. 7(d) shows that there are many cracks vertical with the sliding tracks, and the integrity of coating was poor. The darker areas directed by the arrows were oxides, which were formed during the high temperature tribo-test. The indents generated in the alloy coating surface along the sliding direction were formed by the welding phenomenon, which was caused by imprisoning the fragments between the specimen surface and the pin. Fig. 7(e-f) presents the worn morphologies of Ni-Co-ZrO_2 composite coatings. During the wear test, the ZrO_2 particles carry the load and prevent Ni-Co matrix from wearing by way of dispersive strengthening as discussed in paragraph 1 of section 3.2, and when a large number of ultra-fine ZrO_2 particles were dug out by counter-body from the coating surface. The friction mechanism was transformed from pure sliding to partly rolling, which reducing the friction force and smoothing the counter surface in a similar way by polishing it with ultra-fine particles. Compared with Ni-Co coating, Ni-Co-ZrO_2 composite coatings had the small and slight cracks, it is because ZrO_2

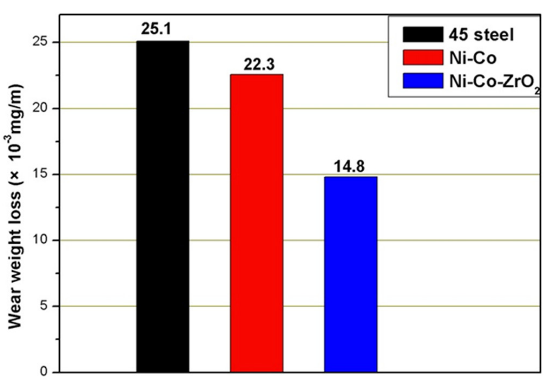


Fig. 4 Wear weight loss of 45 steel, Ni-Co alloy coating and Ni-Co-ZrO2 composite coating at 873 K for 15 min.

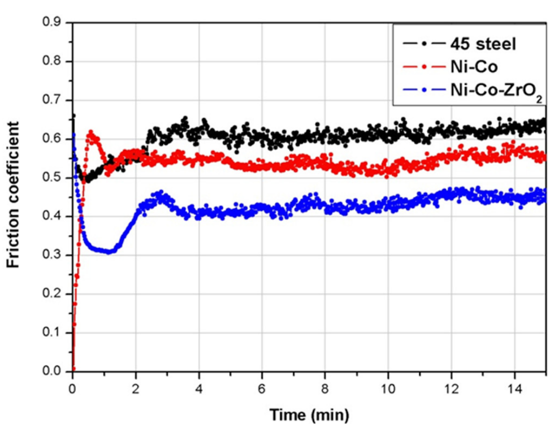


Fig. 5 Friction coefficient of 45 steel substrate, Ni-Co alloy coating and Ni-Co-ZrO2 composite coatings at 873 K.

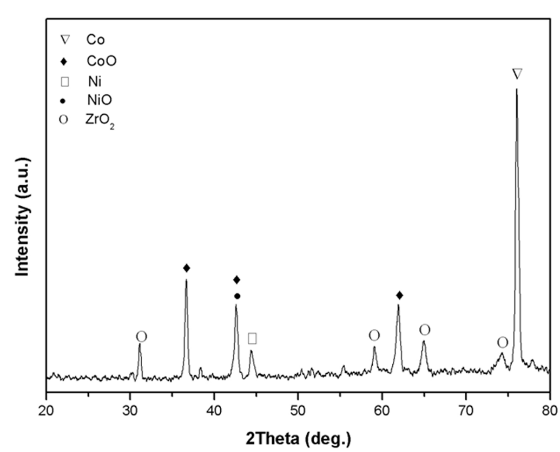


Fig. 6 XRD pattern of Ni-Co-ZrO2 composite coatings after high temperature friction for 15 min at 873 K

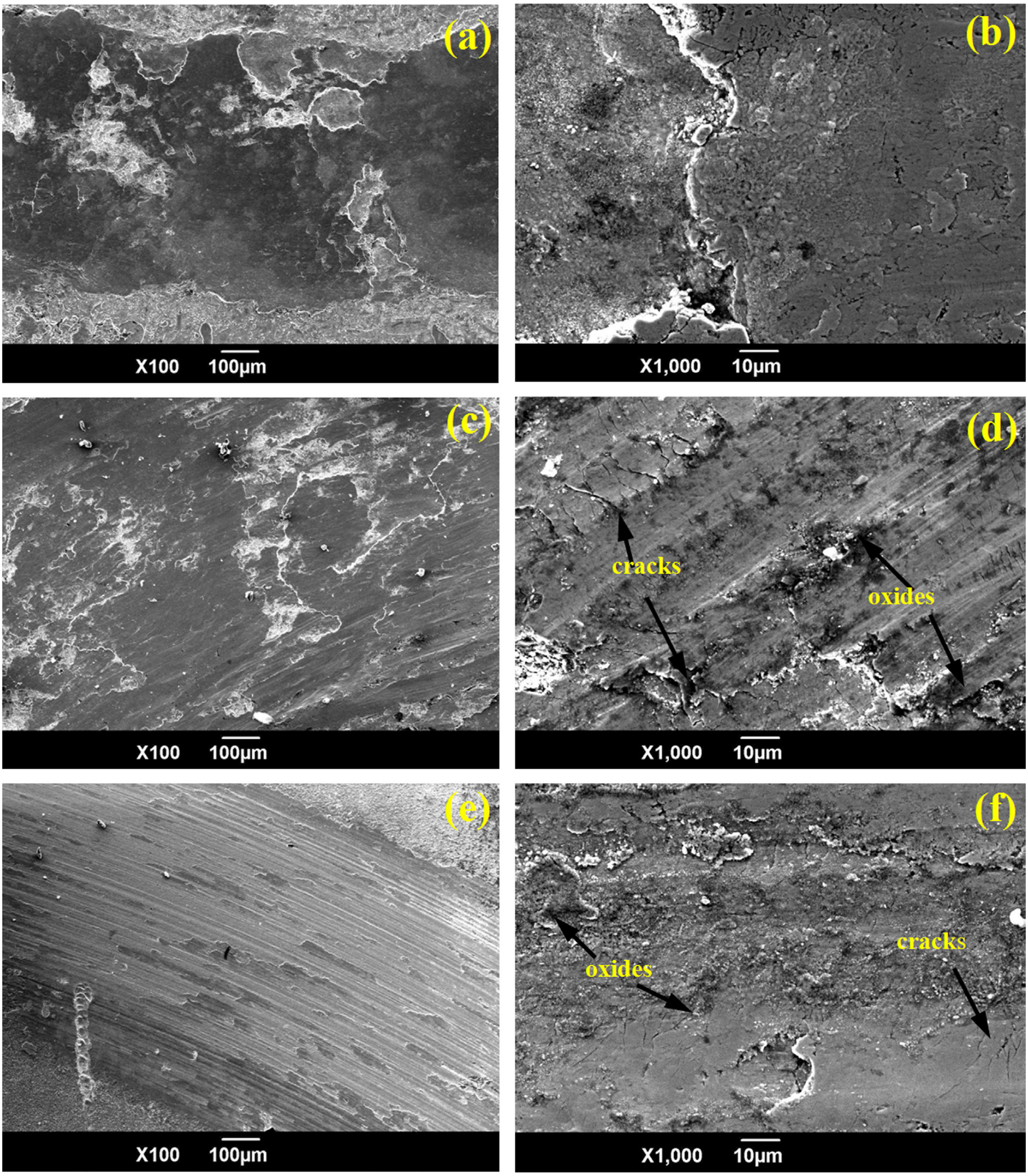


Fig. 7 SEM morphologies of the surface of 45 carbon steel (a-b), Ni-Co alloy coating (c-d) and Ni-Co-ZrO2 composite coating (e-f) after friction with the load of 10 N for 264 m at 873 K.

particles could reinforce composite coating and hinder the growth of the cracks, and hence the friction coefficient became lower, it was also confirmed by Fig. 5. Ni-Co-ZrO_2 composite coating possesses better oxidation resistance than that of Ni-Co coating and 45 steel as discussed earlier, so Ni-Co-ZrO_2 composite coating was not oxidized so heavily as Ni-Co coating and 45 steel. Compared with Fig. 7(a-b) and (c-d), it can be seen from all SEM morphologies that the surface of Ni-Co-ZrO_2 composite coating is the smoothest, the integrity is the best and the oxidation is the lightest. So the Ni-Co-ZrO_2 composite coating possesses higher wear resistance and lower wear rate than that of Ni-Co alloy coating and 45 steel.

3.3 Oxidation resistance

The oxidation experiments were carried out at 573 K, 873 K and 1173 K for 2 h, respectively. Fig. 8 shows the weight gain of 45 carbon steel, Ni-Co alloy coating and Ni-Co-ZrO2 composite coating at different temperatures. From Fig. 8, it can be seen that with the increase of the temperature, the weight gain of 45 steel, Ni-Co alloy coating and Ni-Co-ZrO2 composite coating are all increased. When the oxidation temperature is 873 K, the weight gain of 45 steel, Ni-Co alloy coating and Ni-Co-ZrO2 composite coating is lower. While it is up to 1173 K, the weight gain of 45 steel rapidly increased by 13.4 times, higher than that of Ni-Co coating (9.7 times), and much higher than that of Ni-Co-ZrO2 composite coating (7.8 times). It indicates that Ni-Co-ZrO2 composite coating possesses the best high temperature oxidation resistance.

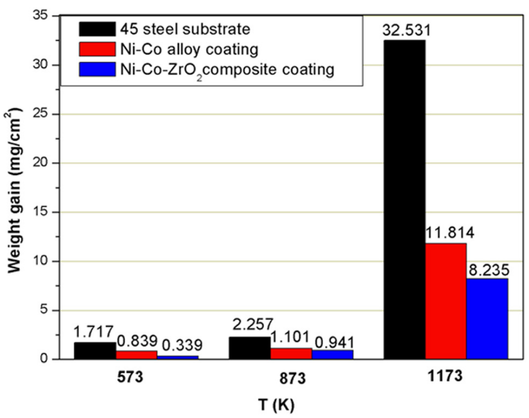


Fig. 8 Weight gain of 45 steel, Ni-Co alloy coating and Ni-Co-ZrO2 composite coating after oxidation test for 2 h at different temperatures.

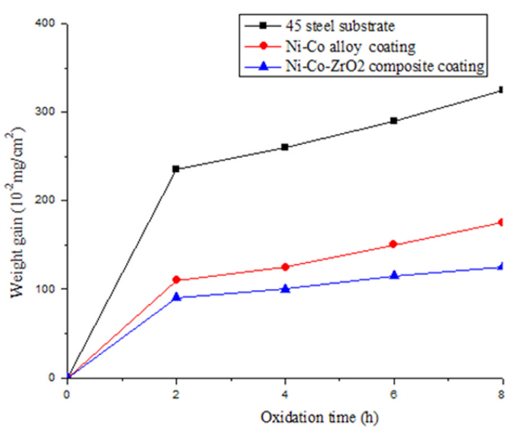


Fig. 9 Weight gain of 45 steel, Ni-Co alloy coating and Ni-Co-ZrO2 composite coating after high temperature oxidation test at 873 K.

Fig. 9 gives the oxidation weight gain curves for 45 steel, Ni-Co coating and Ni-Co-ZrO2 composite coating samples at 873 K. From Fig. 9, it can be seen that the weight gain of 45 steel, Ni-Co alloy coating and Ni-Co-ZrO2 composite coating increase with the increase of oxidation time. Compared with 45 steel, the oxidation rate of Ni-Co coating and Ni-Co-ZrO2 composite coating reduce significantly after 2 h. Meanwhile, the weight gain and the oxidation rate of Ni-Co-ZrO2 composite coating are lower than that of Ni-Co alloy coating, and it means that Ni-Co-ZrO2 composite coating possesses better oxidation resistance than that of Ni-Co coating.

Fig. 10 shows the cross-section micrographs of the Ni-Co coating and Ni-Co-ZrO_2 composite coating after high temperature oxidation test at 873 K for 6 h. It was found that the surface of Ni-Co coating appears black oxide, accompanied by a little crack. In contrast, Ni-Co-ZrO_2 composite coating is still intact, and there are no crack and fall off. And in Fig. 10(b), we can see that the oxide layers obviously. It can be confirmed that the oxide layer of Ni-Co-ZrO_2 composite coating is smooth and uniform in comparison with that of Ni-Co coating after high temperature oxidation test.

Fig. 11 gives EDS analysis of the cross-section micrographs of Ni-Co coating and Ni-Co-ZrO_2 composite coating after high temperature oxidation test at 873 K for 6 h. It shows that the oxide layers contain the elements of Ni, Co and O. Fig. 12 shows the line scanning analysis diagram, suggesting that the oxygen content in the coating increases and the content of Co is still high. It can be demonstrated that the oxide layer has formed on the surface of Ni-Co coating and Ni-Co-ZrO_2 composite coating. However, the content of embedded ZrO_2 particles in the Ni-Co-ZrO_2 composite coating decreases after high temperature oxidation test, the reason why the ZrO_2 particle decreases needs to be further explored.

Fig. 13 shows the SEM micrographs of surface morphology of Ni-Co coating and Ni-Co-ZrO_2 composite coating after high temperature oxidation test. The aggregation particles of Ni-Co composite coating on the surface are uneven, where appears some irregular lumps; however, the particles of Ni-Co-ZrO_2 composite coating on the surface are uniform

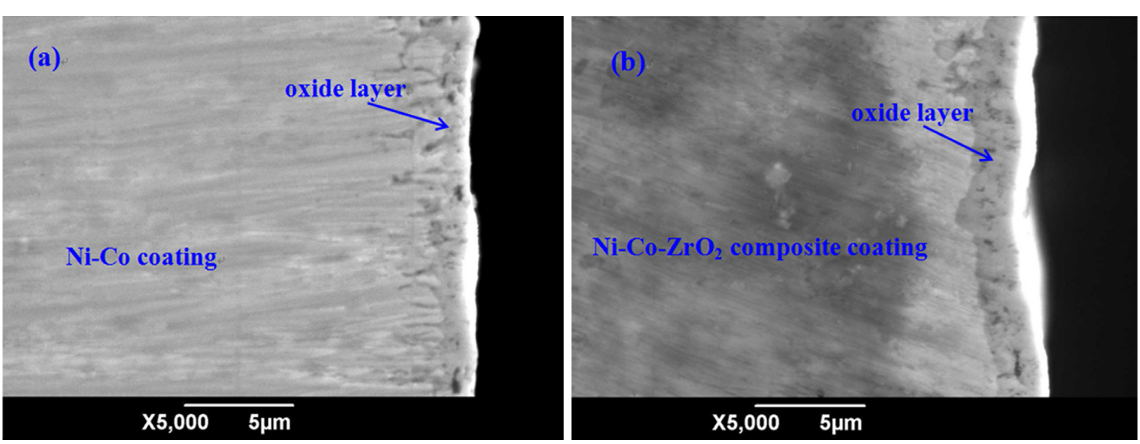


Fig. 10 Cross-section micrographs after high temperature oxidation test for 6 h at 873 K: (a) Ni-Co coating (b) Ni-Co-ZrO2 composite coating

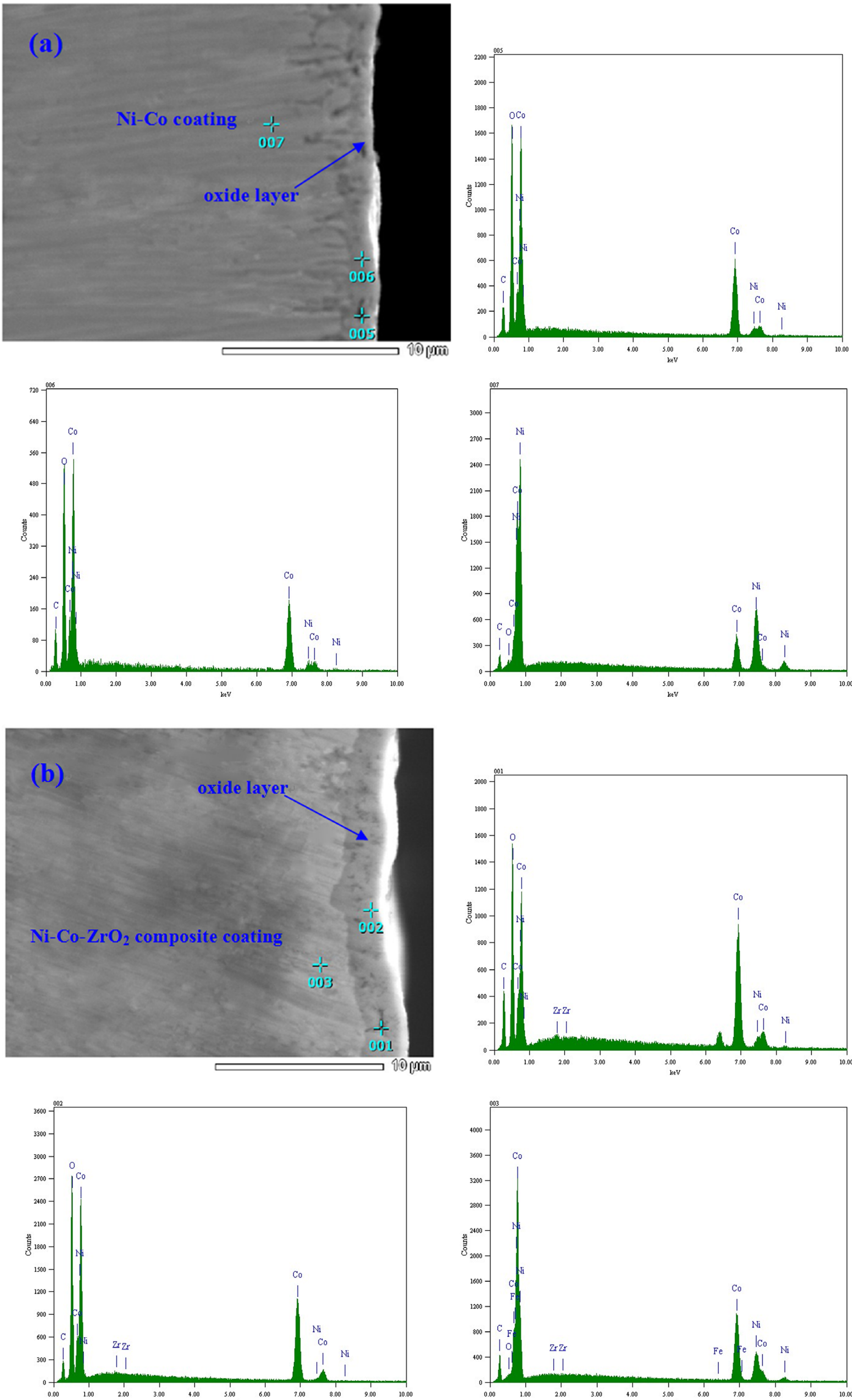


Fig. 11 EDS analysis of the cross-section: (a) Ni-Co coating (b) Ni-Co-ZrO2 composite coating after high temperature oxidation test for 6 h at 873 K.

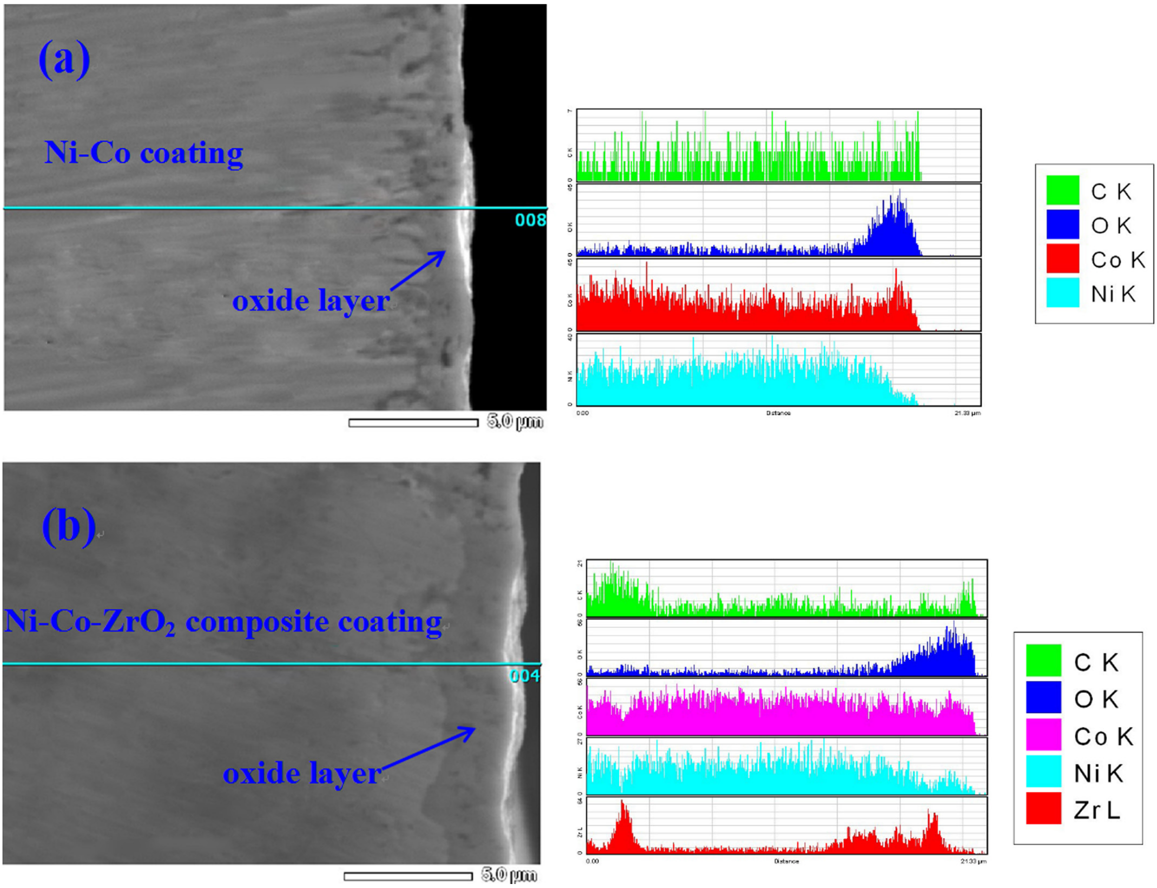


Fig. 12 Line scanning analysis diagram after high temperature oxidation test for 6 h at 873 K (a) Ni-Co coating (b) Ni-Co-ZrO2 composite coating

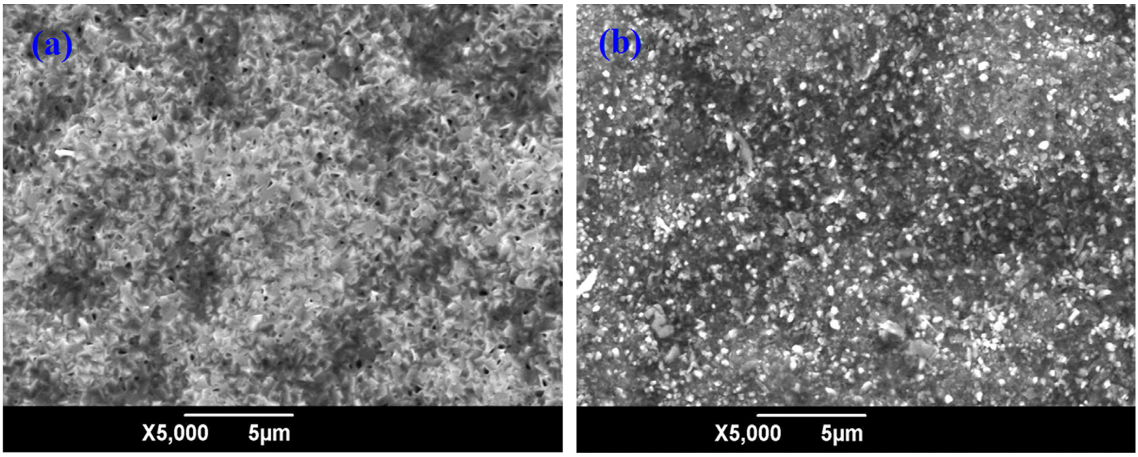


Fig. 13 SEM micrographs of the surface after high temperature oxidation test for 6 h at 873 K (a) Ni-Co coating (b) Ni-Co-ZrO2 composite coating

and tiny, which confirms the analysis of the oxidation weight gain curves of two coatings.

Fig. 14 presents the X-ray diffraction patterns of Ni-Co coating and Ni-Co-ZrO_2 composite coating. Fig. 14(a),(c) show XRD patterns of Ni-Co coating and Ni-Co-ZrO_2 composite coating before high temperature oxidation test, and Fig. 14(b),(d) show XRD patterns of Ni-Co coating and Ni-Co-ZrO_2 composite coating after high temperature oxidation test for 6 h at 873 K. It can be seen that NiO and CoO formed after high temperature oxidation test.

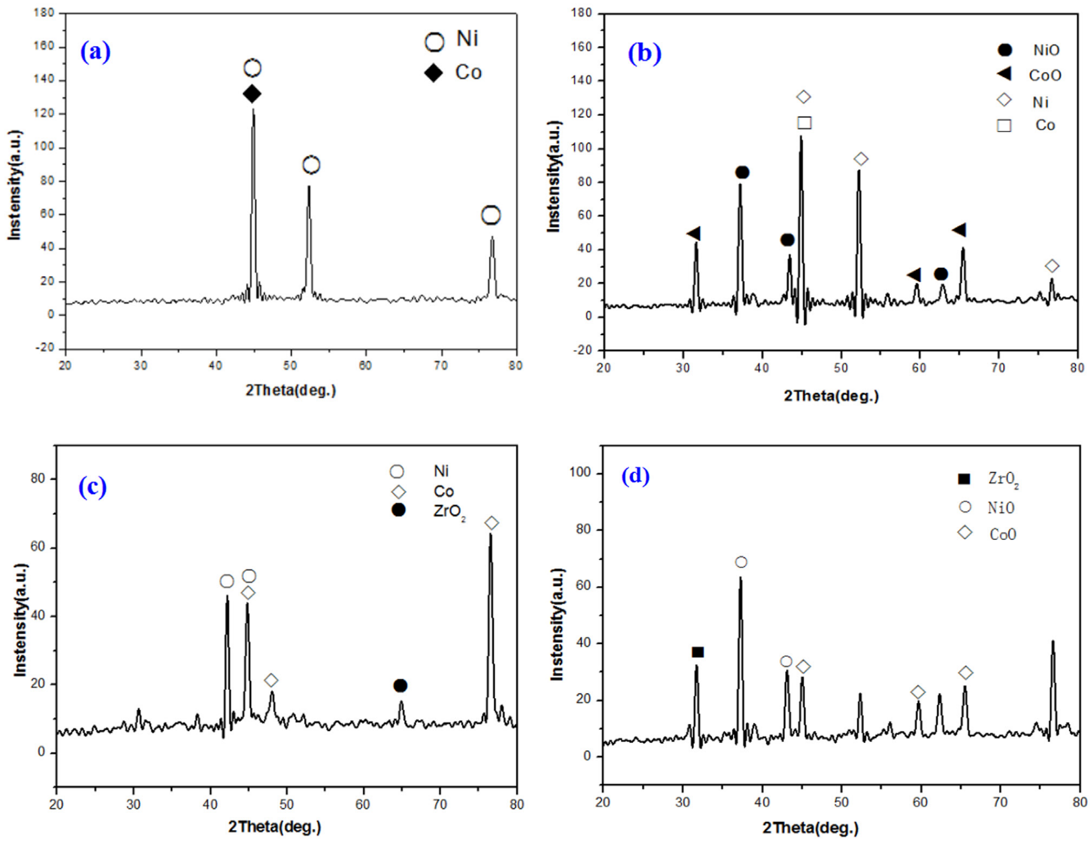


Fig. 14 XRD pattern before and after high temperature oxidation test for 6 h at 873 K (a) (b)of Ni-Co coating (c) (d) Ni-Co-ZrO2 composite coating

In summary, it is well known that the oxygen diffusion through the coating is the dominant diffusion mechanism during the oxidation process [3]. The reason why Ni-Co-ZrO_2 composite coatings can improve the high temperature oxidation resistance is that the dispersive ZrO_2 particles in the Ni-Co matrix reduce the effective area of Ni-Co alloy contacting with ambient oxygen. Furthermore, the ZrO_2 particles exert a reactive-phase effect on the growth of NiO and CoO in the composite coating and effectively reduce the oxidation rate of the composite coating at high temperature. In addition, new phases like NiO and CoO further prevent inward oxygen diffusion. It is also one of the important factors that enable the composite coating to achieve high temperature oxidation resistance.

4. Conclusions

1) Ni-Co alloy and ZrO_2 micron particles were codeposited on 45 steel by electrodeposition and the deposited composite coating shows dispersed ZrO_2 particles and continuous Ni-Co matrix, and there are no pores and cracks and other defects at the interface between the composite coating and the substrate.

2) The embedded ZrO2 particles change the friction mechanism from adhesive wear to abrasive wear. The wear loss rate and coefficient of Ni-Co-ZrO2 composite coating are lower than that of Ni-Co alloy and much lower than that of 45 steel, and the Ni-Co-ZrO2 composite coating possesses the best high temperature wear resistance.

3) The embedded ZrO_2 particles exert a reactive-phase effect on the growth of nickel oxide (NiO) and cobalt oxide (CoO) and effectively reduce the oxidation rate of the substrate at high temperature. The oxidation weight gain of Ni-Co-ZrO_2 composite coating is lower than that of Ni-Co coating, demonstrating that the Ni-Co-ZrO_2 composite coating presents better high temperature oxidation resistance.

Acknowledgments

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