High-temperature Oxidation of Electrodeposited Ni-Co-SiC Composite Coatings

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Abstract. Ni-Co-SiC composite coatings were prepared on 45 carbon steel by electrodeposition for high temperature anti-oxidation. The high-temperature oxidation was studied by circulating oxidation test. The prepared composite coating shows continuous Ni-Co matrix and uniformly dispersed SiC particles, and there are no cracks and pores and other flaws in the composite coating. The results also reveal that due to the reactive-phase effect of the embedded SiC particles, the oxidation rate of the substrate decreases largely at high temperature. The Ni-Co-SiC composite coating exhibits better high-temperature oxidation compared with Ni-Co coating and 45 carbon steel.

1. Introduction

In various engineering applications, the long term performances under high-temperature, high-pressure and high-speed conditions in aggressive solution or atmosphere are crucial to mechanical products and parts, made of carbon steels. For example, major failure for hot forging process is originated from high temperature wear, and proportion of which can reach 70-80% [1]. Moreover, the majority of engines and cutting tools are also damaged due to the reason as just mentioned. For the purpose of improving oxidation and wear resistance at high-temperature, corrosion resistance of engineering materials, surface modification methods are required, in which preparing high-temperature coating is a good choice. Composite coatings incorporated with fine particles, owning satisfying wear resistance and oxidation resistance, which is composed of matrix metal and uniformly dispersed fine ceramic particles such as ZrO2 [2], B4C [3], Si3N4 [4], WC [5], Al2O3 [2,4,6], CeO2 [7], and SiC particles [4,8], among which SiC particles is widely used as the reinforcing particles owing to its high hardness and high-temperature stability. In addition, Ni-Co alloys show brilliant thermal, physical and mechanical properties than pure Ni [9]. Therefore, a novel composite coating with excellent physical and chemical properties, achieved by electrodeposition, is led to by the incorporation of different particles within Ni-Co matrix [6-8]. At present, a large amount of research report concentrate on enhancing the microindentation hardness, wear resistance and corrosion resistance of Ni-Co coatings [6-8,10]. Nevertheless, there is little report about their oxidation behavior.

In our study, Ni-Co thin film incorporated with micron SiC particles which can overcome a lot of restrictions under severe work conditions was made by electrodeposition. The oxidation resistance at high temperature of the three kinds of samples was investigated.
2. Experimental procedures

2.1. Electrodeposition of Ni-Co-SiC coatings
A DC power supply was used in the electrodeposition. A 45 carbon steel substrate (12 mm×10 mm×6 mm) was used as the cathode, and two nickel plates were used as the anode. The SiC particles with an average size of about 3.5 μm were pretreated with 37.5% hydrochloric acid for 6 h and cleaned by distilled water. Before the codeposition, the suspension was treated with ultrasonicitation for 4 h. At the same time, the suspension was stirred by a magnetic stirrer and the temperature of the bath is controlled to be 328 K. For the purpose of obtaining a coating with a thickness of 185 μm, the plating duration was maintained 80 min. Table 1 shows that the process parameters of electrodeposition.

Table 1. Plating bath composition and experimental conditions

| Composition of plating solution [g/L] |  |
|--------------------------------------|---|
| NiSO₄·6H₂O                            | 400 |
| CoSO₄·7H₂O                            | 25  |
| H₃BO₄                                | 30  |
| CH₃(CH₂)₁₁OSO₃Na                      | 0.2 |
| SiC (about 3.5 μm)                    | 80  |

| Experimental conditions              |
|--------------------------------------|
| Temperature                          | 328 [K] |
| pH                                   | 2.5     |
| Current density                      | 5 [A/dm²] |
| Magnetic stirring rate               | 250 [rpm] |
| Plating duration                     | 80 [min] |

2.2. High temperature oxidation tests
We performed oxidation test at high temperature in a muffle furnace with an automatic temperature control device (precision ±2 K). 2 hours of oxidation experiments were performed at 573 K and 1173 K, while at 873K for 2h, 4h, 6h, and 8h, respectively. Subsequently, the oxidation weight increment of the specimens before and after high-temperature oxidation was weighed by using an analytical balance (precision 0.01 mg) and compared with that of the 45 carbon steel and Ni-Co film.

3. Results and discussion
As shown in the insets of Figure 1, the oxidation weight increment of three kinds of specimens at different temperature. We observed that when the temperature increased, the oxidation weight gain of three kinds of samples was also increased. We can observed that the oxidation weight increment of the samples is obvious when the temperature was 1173 k. Compared with 45 steel, both coatings show lower weight gain. And the weight gain of the composite coating is the least at different temperature. This suggested that Ni-Co-SiC coatings would result in less oxide. The relative weight gain at 573K for Ni-Co film and Ni-Co-SiC multilayer films relative to 45 carbon steel is 49% and 6%. The relative weight gain at 873K remains the same for Ni-Co coating at 49%, but increases to 35% for Ni-Co-SiC composite coating. The relative oxidation weight gain decreases to 36% and 26% at 1173K for Ni-Co film and Ni-Co-SiC multilayer films respectively. Therefore, the composite coating exhibits a better high-temperature oxidation resistance.
Figure 1. Oxidation weight increment of 45 carbon steel, Ni-Co film and Ni-Co-SiC multilayer films at different temperature

Figure 2 shows the circulation oxidation weight increment curves of three kinds of samples after heating in the air to 873K. As can be seen from the picture, the three samples had a rapid oxidation weight gain at the first two hours. The reason for the rapid weight gain is the formation of oxide film. At this stage, the weight growth depends mainly on the oxidation reaction, rather than the atomic diffusion. In the meantime, we can also see from the image that the mass gain and oxidation rate of the Ni-Co-SiC multilayer films were the smallest. After two hours oxidation, the coatings surface was oxidized. And the oxide layer becomes more and more compact and thicker. So the mass gain of the coated samples decreased obviously. What is more, the compact oxide layer turns to a barrier for oxygen diffusion, and the inner metal alloy was protected by the oxide layer, so that the oxidation rate of multilayer films and composite coating decreases after 2 h. In the meantime, the mass gain of the Ni-Co-SiC multilayer films is less than that of the Ni-Co film. This is due to the formation of a protective oxide layer on the surface of the film, hindering the diffusion of oxygen, and reduced the oxidation rate. Thus, the Ni-Co-SiC multilayer films exhibit a better oxidation resistance at high temperature.

The experimental results show that the oxidation weight increment of the Ni-Co-SiC composite coatings is obviously reduced. The high temperature oxidation resistance of the Ni-Co-SiC multilayer films is improved because of the SiC particles with high thermal stability are dispersed in the Ni-Co matrix. Therefore, it is easy to form an oxide film with fine grain and uniformly compact structure. SiC particles can not decompose at high temperatures, but uniformly dispersed in the Ni-Co matrix. These fine particles dispersed in the oxide film, inhibiting the growth of the film and reducing the grain size, leading to a compact oxide layer. The formation of a dense protective layer can effectively prevent the further reaction of the matrix and oxygen and reduce the oxidation rate. The compactness of the Ni-Co-SiC multilayer films was significantly improved, the porosity was reduced, and the internal diffusion rate of oxygen in the coating was slowed down at the initial stage of oxidation, which directly reduced the oxidation rate, and also reduced the oxidation weight gain.
Figure 2. Oxidation weight increment of 45 carbon steel, Ni-Co film and Ni-Co-SiC multilayer films at 873 K with different oxidation time

4. Conclusions
Ni-Co-SiC multilayer films were prepared on 45 carbon steel substrate by electrodeposition. The prepared composite coating shows continuous Ni-Co matrix and uniformly dispersed SiC particles, and there are no cracks and pores and other flaws in the composite coating. Due to the reactive-phase effect of the embedded SiC particles, the oxidation weight increment of Ni-Co-SiC multilayer films is lower than that of Ni-Co film and 45 carbon steel. Therefore the Ni-Co-SiC coatings exhibit an excellent oxidation resistance at high temperature.

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