



# Effects of aging treatment on microstructure and tribological properties of nickel-based high-temperature self-lubrication wear resistant composite coatings by laser cladding



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## HIGHLIGHTS

- The composite coating had excellent high-temperature (600 °C) phase stability.
- The average microhardness of the aged coating was slightly decreased.
- The tribological properties were not significantly affected by aging treatment.

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## ABSTRACT

The NiCr/Cr<sub>3</sub>C<sub>2</sub>-WS<sub>2</sub> high-temperature self-lubrication wear resistant composite coatings were fabricated on substrate of a hot-rolled AISI304 austenitic stainless steel by laser cladding. The high-temperature phase stability of the composite coatings was evaluated by aging at 600 °C for 10 h, 30 h, 50 h, and the microstructures of the as-laser clad and aged coatings were examined by means of XRD, SEM, EDS, respectively. The sliding wear resistance of the as-laser clad and aged coatings was evaluated at 600 °C. The results show that NiCr/Cr<sub>3</sub>C<sub>2</sub>-WS<sub>2</sub> composite coating has excellent high-temperature phase stability, the  $\gamma$ -(Fe,Ni)/Cr<sub>7</sub>C<sub>3</sub> eutectic phases, Cr<sub>7</sub>C<sub>3</sub> and (Cr,W)C hard phases, CrS/WS<sub>2</sub> mixed solid lubricant phases all existed in the as-laser clad and aged coatings. The volume fraction of eutectic phases decreased gradually with the increasing of aged time due to their dissolution. The microhardness of the aged coating decreased slightly after aging the coating 50 h at 600 °C due to the dissolution of the eutectic phases and notable breaking or granulation of the Cr<sub>7</sub>C<sub>3</sub> hard phase, but the tribological properties were not significantly affected by aging treatment.

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## 1. Introduction

In advanced industrial gas turbines, the power generation industries and aeronautical as well as chemical industries, there are many tribological components working under high temperature and aggressive service conditions [1,2], demanding novel corrosion and high-temperature oxidation resistant materials, which also have outstanding antifriction [3] and self-lubrication performance [4]. In this regard, surface modification techniques offer a solution to the problem. Among the various techniques, laser cladding is especially attractive because of its unique features in the process

control. Laser cladding is the fusion of an alloy powder on a substrate and admits the melting of substrate occurred only within a very thin layer [5,6]. The laser energy melts the cladding material forming a metallurgical bond with the substrate [7–9]. Laser cladding NiCr–Cr<sub>3</sub>C<sub>2</sub> cermet coatings have been widely used for high temperature wear and corrosion resistance applications in various industrial applications [10,11]. However, due to the high sliding friction coefficient and high hardness of NiCr–Cr<sub>3</sub>C<sub>2</sub> coating, the counter materials sliding against NiCr–Cr<sub>3</sub>C<sub>2</sub> coating exhibit significantly high wear rates, which limits their application on moving components, such as rotating devices and other places [12,13]. In order to reduce the friction coefficient and improve the tribological properties of this kind of wear-resistant hard coatings, WS<sub>2</sub>, which has the lamellar structure like MoS<sub>2</sub> and graphite, is easy to be sheared and form transfer lubricious films between the

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friction pair interface, has been employed as solid lubricant in numerous studies [14,15]. In our previous study, the NiCr/Cr<sub>3</sub>C<sub>2</sub>–WS<sub>2</sub> alloy system was designed and fabricated by the laser cladding process and exhibited excellent wear resistance under both room and high-temperature sliding wear test conditions [16].

The long-term high-temperature phase stability is critically important for materials in high-temperature environments, especially for a laser clad coating with a rapidly solidified non-equilibrium microstructure. This is because, during long-term elevated-temperature exposure, unfavorable phase transformation and serious elemental diffusion from coating into the substrate are two of the most important reasons leading to degradation of a high-temperature coating [17]. G.D Shi et al. [18] found that the Ni<sub>3</sub>Al phase appears in the NiCoCrAl alloy sheet after heat treatment which is favorable to improve the interface bonding between the columnar structures. The residual stress in the NiCoCrAl alloy sheet after heat treatment is reduced significantly. C.X. Yang et al. [19] studied the effects of heat treatments on microstructure and mechanical properties of Rene 80. Consequently, it is very important to improve the mechanical properties of Rene 80. J.S.C. Jang et al. [20] found that the effect of heat treatment exhibited significant improvement on the ductility of the Ni–19Si–3Nb–0.15B alloy. But to the best knowledge of the authors, the effects of aging treatment on microstructure and tribological properties of NiCr/Cr<sub>3</sub>C<sub>2</sub>–WS<sub>2</sub> alloy system have rarely been reported in open literature.

In this paper, the high-temperature phase stability of NiCr/Cr<sub>3</sub>C<sub>2</sub>–WS<sub>2</sub> high-temperature self-lubrication wear resistant composite coating was evaluated by aging at 600 °C for 10 h, 30 h, 50 h, respectively in ambient air. The effects of aging treatment on microstructure and wear resistance of the coating were investigated as a function of aging time, and the corresponding mechanisms were also discussed.

## 2. Experimental procedures

The substrate used in this study was a hot-rolled AISI304 austenitic stainless steel with a chemical composition of Fe–0.07C–1.0Si–2.0Mn–18Cr–9Ni (wt.%) and rectangular dimensions of 50 × 40 × 8 mm<sup>3</sup>. Before laser cladding, the working surface of the stainless steel was ground to a surface roughness of Ra = 0.2 μm and rinsed with ethanol and acetone. The mixtures of Ni<sub>80</sub>Cr<sub>20</sub>–Cr<sub>3</sub>C<sub>2</sub> (70)(wt.%) with average particle diameter less than 45 μm and 10%WS<sub>2</sub> were mixed by using a ball miller and used as the pre-placed material. The composite powders were mixed with cellulose acetate, pasted onto the substrate and dried in an oven at 80 °C for 2 h. The thickness of the pastes was approximately 1.0 mm.

Laser cladding was conducted on a GS-TFL-10 kW continuous wave CO<sub>2</sub> laser system equipped with a five-axis computer numerical controlled (CNC) platform. Nitrogen gas was used to protect the surface from oxidation. The optimized laser processing parameters were: laser beam output power of 1.5 kW, beam size of 6 mm × 3 mm and beam scanning speed of 6 mm s<sup>−1</sup>.

Metallographic and wear test specimens were prepared by electric discharging machining (EDM) followed by mechanical milling and grinding. Metallographic sections were prepared using standard mechanical polishing procedures and were etched in HCl:HNO<sub>3</sub> solution in volume ratio of 3:1 at room temperature for approximately 30 s. Microstructure was characterized using KYKY-EM3200 and S-4700 scanning electron microscope (SEM) incorporating energy dispersive X-ray spectroscopy (EDS) analysis and operated at 10 kV. Phase constituents of the coatings were identified by X-ray diffractometer (XRD) conducted by the Pert-Pro MPD X-ray diffractometer with Cu target K $\alpha$  radiation operated at 40 kV and 40 mV. The microhardness of the cross section from the coating

**Table 1**  
Experimental parameters of wear test.

Load (N)	Temperature (°C)	Wear time (min)	Rotation radius (mm)	Linear velocity (m min <sup>−1</sup> )
5	600	20	2	16.889

surface to the substrate was measured using a MH-5 micro-hardness tester with a load of 300 g and dwell time of 10 s.

The aging temperature was selected as 600 °C, because the fabricated composite coatings on austenitic stainless steel are expected to be used in circumstance no more than 600 °C. The high-temperature phase stability of the coating was evaluated by aging the coating at 600 °C for 10 h, 30 h, 50 h, respectively in ambient air.

Dry sliding wear tests of the high-temperature wear resistance of the as-laser clad and aged coatings were conducted with a ball-on-disk tribometer (HT-1000 tester, Lanzhou Zhongkekaihua science and technology Co., Ltd., China) in ambient air at 600 °C. During wear testing, the specimen was fixed and rotated following the disk. The sliding wear testing counterpart was very hard ceramic Si<sub>3</sub>N<sub>4</sub> ball with a diameter of 3 mm and hardness of 16 GPa. The selected wear testing parameters were listed in Table 1.

After wear tests, the worn surfaces were rinsed with ethanol and dried, morphologies and the chemical compositions of the worn surfaces were characterized by SEM/EDS. The wear rate was defined as:

$$W = \frac{V}{LS} \quad (1)$$

In which:  $W$  is wear rate;  $V$  is wear volume (mm<sup>3</sup>);  $L$  is load (N);  $S$  is sliding distance (m).

## 3. Results and discussion

### 3.1. Compositions, microstructure and hardness of the coatings

Fig. 1 presents the XRD patterns of the as-laser clad and the aged NiCr/Cr<sub>3</sub>C<sub>2</sub>–WS<sub>2</sub> coatings at 600 °C for 50 h. Combining the results of XRD and EDS analysis, it is indicated that the  $\gamma$ -(Fe,Ni)/Cr<sub>7</sub>C<sub>3</sub> eutectic phases, Cr<sub>7</sub>C<sub>3</sub> and (Cr,W)C hard phases, CrS/WS<sub>2</sub> mixed solid lubricant phases all exist in the as-laser clad and the aged NiCr/Cr<sub>3</sub>C<sub>2</sub>–WS<sub>2</sub> coating at 600 °C for 50 h. As shown clearly in Fig. 1(b), the coating aged at 600 °C for 50 h produced no evident changes on phase constituents. It indicated that the laser cladding NiCr/Cr<sub>3</sub>C<sub>2</sub>–WS<sub>2</sub> self-lubrication wear resistant composite coating has excellent high-temperature phase stability, which is beneficial for the coating to be applied as high-temperature tribological coatings at service temperatures near 600 °C.

Fig. 2 shows the typical microstructure of the as-clad NiCr/Cr<sub>3</sub>C<sub>2</sub>–WS<sub>2</sub> coating. The as-clad NiCr/Cr<sub>3</sub>C<sub>2</sub>–WS<sub>2</sub> coating has a fine and uniform microstructure and is metallurgically bonded to the substrate with no pores and no cracks, as shown in Fig. 2(a). The element content of each phase in Fig. 2(b) was analyzed by EDS and relevant XRD patterns. The large blocky phases were identified as hard Cr<sub>7</sub>C<sub>3</sub> and (Cr,W)C carbides, while the gray spherical or granular patches were recognized as CrS and WS<sub>2</sub> sulfides, which are expected to possess solid lubricant effect. Most of premixed WS<sub>2</sub> is decomposed and oxidized, its microstructure is not easy to be observed, but can be inferred from XRD results. The colony phase was identified as  $\gamma$ -(Fe,Ni)/Cr<sub>7</sub>C<sub>3</sub> eutectic phase. According to the report of Liu et al. [21], with the rapid solidification of the melt pool, the Cr<sub>7</sub>C<sub>3</sub> phase was primary formed attributed to its highest melting point (1838 K) and lowest Gibbs free energy. Then the

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