Corrosion Behavior of a Ni–Cr–Mo Alloy Coating Fabricated by Laser Cladding in a Simulated Sulfuric Acid Dew Point Corrosion Environment

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Abstract: For this study, aimed at proposing a potential direction to prevent sulfuric acid dew point corrosion, a Ni–Cr–Mo alloy Hastelloy C22 coating was fabricated by coaxial laser cladding technology. The phase composition, microstructure, and corrosion behavior in a simulated sulfuric acid dew point corrosion environment were investigated and compared with a Hastelloy C22 alloy, a titanium alloy TC4, and 09CrCuSb steel (ND). The results showed that the phase composition of the C22 coating is essentially similar to that of the C22 alloy, consisting of a $\gamma$-Ni solid solution and Ni6Mo6C1.06. The finer microstructure of the C22 coating mainly contains eutectic and dentrite, presenting a typical solidification feature of laser cladding. The corrosion resistance of the C22 coating is very close to that of the C22 alloy, and outclasses that of TC4 and ND. The corrosion behavior of the C22 coating is intergranular corrosion resulting from the segregation of molybdenum, chromium containing corrosion products, and smaller anode micro-batteries.

Keywords: laser cladding; Ni–Cr–Mo alloy coating; corrosion behavior; sulfuric acid dew point corrosion

1. Introduction

Sulfuric acid dew point corrosion caused by low-temperature flue gas exists extensively in the backend of coal-fired boilers and gives rise to the severe failure of components [1–6]. As the temperature of the flue gas decreases to 200 ºC, 99% of the SO$_3$ in the flue gas combines with water vapor to form sulfuric acid vapor. After contacting with the low-temperature surfaces of devices, the sulfuric acid vapor in the flue gas condenses into sulfuric acid droplets because of the temperature’s sharp decrease [7–10]. Serious corrosion caused by concentrated sulfuric acid in low temperature (<200 ºC) is defined as sulfuric acid dew point corrosion, which is considered as the major reason of the corrosion failure of low-temperature devices [11–15]. More importantly, because the limestone–gypsum wet flue gas desulfurization (WFGD) system is used worldwide, especially in 90% the power plants in China [16–19], the flue gas is sharply cooled down to only 50 ºC or lower and mixed with a larger amount of water vapor and droplets in the WFGD system. Therefore, the condensation of sulfuric acid and consequential sulfuric acid dew point corrosion are unavoidable [1,20–24]. Employing anti-corrosion materials and surface treatment technology is considered as an effective solution from the standpoint of economic efficiency and complete reliability of the facilities [24–27].

Anti-corrosion materials widely employed in the low-temperature surface of boilers mainly include austenite and duplex stainless steels, titanium, and nickel-based alloys [28–33]. High performance nickel-based alloys have been utilized in preheater, FGD systems, backend ductworks, etc., to extend the service life to several decades. Among the nickel-based alloys, the Ni–Cr–Mo alloy is the most...
widely used because of its excellent property in preventing acid corrosion, pitting, and crevice corrosion [13,34–41]. Moreover, the corrosion behavior of the Ni–Cr–Mo alloy has been widely studied. G. Bellanger et al. [39] studied the passivation behavior of C22 in a sulphate solution at pH 3, and found that the increase in temperature attacked the passivity. As is widely known, laser cladding coating technology is an effective measure to obtain excellent surface properties because of confirmed advantages such as high energy density, metallurgical bonding, low dilution rate, and small heat affected zone [42–45]. Furthermore, various laser cladding Ni–Cr–Mo alloy coatings show excellent corrosion resistance in several conventional corrosive solutions at room temperature [46–53]. Q.Y. Wang et al. [47–49] studied the characteristics and corrosion resistance of laser cladding C22 coatings, and obtained performance-enhancing methods including cladding parameter optimization, remelting, and mechanical polishing. L. Chen et al. [51] studied corrosion behavior under the multi-factor impingement of a Hastelloy C22 coating in a 3.5% NaCl solution. However, the corrosion behavior of a laser cladding Ni–Cr–Mo alloy coating in a simulated sulfuric acid dew point environment has been barely studied, which will be valuable in developing a new promising corrosion-preventing technology for coal-fired boilers.

Coaxial laser cladding technology has attracted much attention because of its excellent performance regarding dilution, powder utilization, and processing efficiency [54–57]. This technology was applied to fabricate the Hastelloy C22 coating, and the sulfuric acid dew point corrosion behavior of the C22 coating was investigated in the current work. The laboratory immersion corrosion testing in sulfuric acid at different temperatures was conducted to simulate a sulfuric acid dew point corrosion environment. A comparison of corrosion resistance was made among the Hastelloy C22 coating, the cast Hastelloy C22 alloy, the titanium alloy TC4, and 09CrCuSb (ND steel), the last three of which are widely employed as anti-corrosion materials in sulfuric acid dew point environments [20,23,58,59]. The experimental results are discussed thoroughly, and the corresponding corrosion behavior of the laser cladding Hastelloy C22 coating is presented in detail.

2. Materials and Methods

2.1. Materials

The chemical composition of the Hastelloy C22 powders used for cladding C22 coatings is listed in Table 1, along with the cast Hastelloy C22 bulk, TC4 bulk, and 09CrCuSb (ND steel) bulk. The Hastelloy C22 powders with a diameter in the range of 46-150 µm were offered by Beijing General Research Institute of Mining and Metallurgy (BGRIMM), Beijing, China. The substrate for cladding process was Q235 steel, which was typical structural material.

| Element | C22 powders | C22 bulk | TC4 | ND | Q235 |
|---------|-------------|----------|-----|----|------|
| Ni      | 21.3        | Bal.     | -   | -  | -    |
| Cr      | 13.2        | 20.0     | 0.92| 0.28| -    |
| Mo      | -           | 13.8     | 0.08| -  | -    |
| Mn      | 0.45        | 0.08     | 0.43| Bal.| -    |
| Fe      | 2.93        | 5.0      | 0.15| 0.28| 0.37 |
| Si      | -           | 1.83     | 0.15| -  | -    |
| Co      | 2.0         | 3.2      | -   | -  | 0.38 |
| V       | -           | -        | -   | -  | 0.08 |
| W       | 3.0         | 0.3      | -   | -  | -    |
| Ti      | -           | -        | -   | -  | -    |
| Al      | -           | 3.8      | -   | 3.8| -    |
| Cu      | -           | Bal.     | -   | -  | -    |
| Sb      | -           | 3.8      | 0.15| -  | -    |
| C       | -           | 0.08     | -   | -  | -    |

2.2. Laser Cladding Process

The coaxial laser cladding process performed in the current work employed a high-power fiber laser (ZKZM-2000, zKzM Laser Technology Co., Ltd., Xi’an, China) and a self-designed coaxial powder feed nozzle. A high-power fiber laser produces a collimated laser beam with a maximum 2000 W power and a beam spot of 1.4 mm in diameter. The coaxial powder feed nozzle was designed to eject three powder streams from uniformly distributed directions around the laser beam, and the powder streams were formed by argon carrier gas with a flow rate of 5 L/min. In order to obtain a homogenous C22 coating that was thick enough, without the influence of diffusion, a multi-track and multilayer
cladding process was adopted to prepare 5 mm thick coating specimens. A schematic of the multi-track coaxial laser cladding process is shown in Figure 1. The optimum laser parameter was determined by the preliminary experiments, with a laser power of 2000 W, an overlap ratio of 40%, and a laser scan speed of 8 mm/s.

![Schematic of the multi-track coaxial laser cladding process.](image)

**Figure 1.** Schematic of the multi-track coaxial laser cladding process.

### 2.3. Characterization Method

The phase composition, microstructure, and element distribution of the prepared C22 coating were analyzed by an X-ray diffractometer (Rigaku D/Max-2400, Tokyo, Japan), a scanning electron microscope (ZEISS EVO 18, Berlin, Germany), and an energy dispersive X-ray spectrometer (Bruker, Billerica, MA, USA), respectively.

### 2.4. Corrosion Experiment Method

The laboratory immersion testing designed according to ASTM G1-03 and ASTM G31-2012 was performed to study the corrosion resistance of the prepared C22 coating in a simulated sulfuric acid dew point corrosion environment [60,61]. To simulate the harsh environment in FDG systems and backend ductworks, a 50 wt.% H$_2$SO$_4$ solution (prepared by analytical grade 98 wt.% H$_2$SO$_4$ and deionized water) at 50, 60, and 70 °C was selected as testing conditions, which are considered to cause the most serious corrosion [19,40,62–64].

The prepared C22 coating was individually cut into slice specimens with dimensions of 20 mm × 10 mm × 3 mm using a wire cutting machine. The cast Hastelloy C22 and TC4 specimens had the same dimensions, but the ND specimen’s dimensions were 20 mm × 10 mm × 2 mm because of the raw material dimensional limit. Pretreatment and cleaning processes following the guidelines stated in ASTM G31-2012 [61]. Triplicate test specimens for each condition were applied to reduce measurement error and occasional exceptions. The initial dimensions of the specimens were measured carefully using a Vernier caliper, and the initial mass was measured using an electronic balance.

The duration of immersion testing under each condition was 12 h a corrosion cycle up to 72 h, and specimens were taken out every 12 h cycle to measure the mass. Before mass measurement, the corrosion products were removed using the chemical cleaning method specified in ASTM G1-03 and ASTM G31-2012 [60,61]. The corrosion product remover solution for the C22 coating, cast C22,
and TC4 was 15 vol.% HCl solution (prepared by analytical grade 98 wt.% hydrochloric acid and deionized water), and the one for ND was a mixture solution of 500 mL 37 wt.% hydrochloric acid +500 mL deionized water +3.5 g hexamethylenetetramine. The corrosion rate was employed to evaluate the corrosion resistance, calculated according to the following formula [61]:

$$R = 8.76 \times 10^4 \times (M - M_1) / (S \times T \times D)$$

(1)

where $R$ is the corrosion rate in mm/a (mm/year), $M$ is the initial mass of the specimen in g, $M_1$ is the mass of the specimen after exposure in g, $S$ is the area of the specimen before exposure in cm$^2$, $T$ is the time of exposure in hours, and $D$ is the density in g/cm$^3$, 8.69 g/cm$^3$ for C22 bulk and the C22 coating, 4.43 g/cm$^3$ for TC4, and 7.8 g/cm$^3$ for ND.

In order to study the corrosion behavior, the corrosion morphology and composition changes were observed through an FESEM device (FEI Quanta 200F, Amsterdam, Netherlands) equipped with EDS (EDAX, New York, NY, USA).

3. Results and Discussion

3.1. XRD Analysis

The X-ray diffraction patterns of the cast Hastelloy C22 bulk and laser cladding C22 coating are shown in Figure 2. It can be observed that the main phase composition of the two specimens is similar, containing Ni-Cr-Co-Mo, (Fe, Ni), Cr$_{0.19}$Fe$_{0.7}$Ni$_{0.11}$, and Fe$_{0.64}$Ni$_{0.3}$. All these phases are a solid solution between Ni and other elements, like Cr, Mo, Fe, etc., constituting a $\gamma$-Ni solid solution as the prime phase of C22 bulk and the C22 coating. It implies that the coaxial laser cladding barely has an impact on the phase composition, resulting in the equally excellent properties as initial materials [65–67]. Nevertheless, there is a difference in the phase content between C22 bulk and the C22 coating according to the diffraction peak intensity of the two specimens. The content of Cr$_{0.19}$Fe$_{0.7}$Ni$_{0.11}$ and Fe$_{0.64}$Ni$_{0.3}$ in the C22 coating is slightly more than that in C22 bulk, probably due to more elemental Fe diffused from the substrate during the laser cladding process. Moreover, the type of carbide in the two specimens is different, i.e., NiC$_x$ in C22 bulk and Ni$_6$Mo$_6$C$_{1.06}$ in the C22 coating. It is assumed that the formation of molybdenum carbide results from elemental Mo separating out during the rapid solidification of the laser cladding [47,51,65]. Similarly, the grain refinement effect arises from the rapid cooling rate of the laser cladding, which is represented by the larger full width at half maximum in the XRD patterns of the C22 coating.

![Figure 2. XRD patterns of C22 bulk and the C22 coating.](image-url)
3.2. SEM and EDS Analysis

The SEM micrograph and element diffusion curve collected by EDS linear scanning of an overall cross-section of the C22 coating is shown in Figure 3. It can be seen that the metallurgical bonding between the light-colored coating and the dark substrate is achieved with a clear and regular interface. The bottom region of the coating bonding to the substrate is darker than the upper region. It is assumed that the larger degree of element diffusion in the first 1 or 2 layers during the multilayer cladding process leads to the color difference. The state of element diffusion is specifically described by the curve in Figure 3b, in which a 1 mm thick diffusion layer between the substrate and the C22 coating is presented. The existence of the diffusion layer and the distribution of Fe can prove that metallurgical bonding was achieved [41–43].

The microstructure of the C22 coating cross-section perpendicular to the laser scanning direction is shown in Figure 4. The wavy interface and floating up of substrate materials are clearly presented in Figure 4a, resulting from the Gaussian distribution of laser beam energy and element diffusion during repeated heating of the overlap region [49,68,69]. Meanwhile, thin planar crystals solidifying parallel to the interface are observed in Figure 4b. This kind of interface morphology provides an excellent binding force between the coating and the substrate. The microstructure of the C22 coating is mainly composed of gray dendrites and bright eutectics forming on the grain boundaries. The large constitutional supercooling and rapid cooling lead to the trapping of element diffusion, thus eutectics with different element compositions form on the boundary of the gray primary dendrites and result in the microstructure with intercrystalline segregation [47,49,65]. Meanwhile, the distribution of solidification is mainly cellular, columnar, and equiaxed in turn from the bottom to the top. In the bottom region, as shown in Figure 4b, cellular solidification occurred above the planar crystals, with a direction perpendicular to the interface. The columnar solidification of the dendrites dominates in the middle region with two growth directions as perpendicular and about 60° to the interface, illustrated in Figure 4a,c. In the top region shown in Figure 4d,e, the solidification of the dendrites transforms to an equiaxed type, accompanied by a decrease in dimension. The amounts of black micropores mainly distribute in the eutectic networks, probably due to the shrinkage defects and ablation that occurred during the laser cladding process.

It is widely recognized that the morphology of the laser cladding coating mainly depends on the G/R ratio during solidification, in which G and R represent temperature gradient and solidification rate, respectively. Overall, the temperature gradient gradually increased from the interface to the top region of the coating during the solidification process. In the region of the interface, the large G caused by the substrate and barely constitutional supercooling resulted in the dominance of planar solidification. With the increase in distance from the interface, the G/R ratio decreased and the constitutional supercooling gradually increased in the molten pool, leading to the transformation into cellular crystals and dendrites. In the molten pool, the direction of solidification tended to go along with the direction of the largest thermal flux density. Therefore, the direction of most cellular and dendritic solidification in the bottom and middle region of the coating was perpendicular to the interface, due to the heat being mainly transmitted from the molten pool to the substrate [43,49,51,52]. The generation of columnar dendrites at about 60° to the interface in the middle region resulted from the combined effect of remelting and resolidification in the overlap region [47,51]. In the top region, the transformation to the finer equiaxed dendrites was attributed to the multi-directional heat transmission and larger constitutional supercooling. Meanwhile, these factors resulted in the trapping of element diffusion, and led to the formation of black micro pores in the bright eutectic networks.

The element distribution mapping of the C22 coating and the elemental content analysis of each phase are shown in Figure 5. It can be clearly seen that the nickel content is dominant in either grains (Point 1) or eutectics (Point 2), and slightly higher within grains. In comparison, elemental molybdenum is found mainly accumulating in the eutectic networks, and a small amount of elemental carbon exists in the eutectic networks and adjacent region. Moreover, the remaining elements, such as chromium, iron, tungsten, and carbon, all distribute uniformly. It is inferred that both primary grains
and secondary eutectics mainly consist of a \( \gamma \)-Ni solid solution, and the main distinction is the amount and type of solute elements. It is notable that the higher content Fe compared to C22 bulk is assumed to have a negative effect on the corrosion resistance of the C22 coating [35–38]. Meanwhile, the black micropores are corroded preferentially and become the nucleation of localized corrosion, leading to the degradation of corrosion resistance.

**Figure 3.** (a) SEM micrograph and (b) element diffusion curve of an overall cross-section of the C22 coating.

**Figure 4.** SEM images of (a) the bottom region at low magnification; (b) the bottom region at high magnification; (c) the middle region; (d) the top region at low magnification; and (e) the top region at high magnification of the laser cladding C22 coating.
3.3. Corrosion Rate Analysis

Corrosion rate (R) versus time curve of the C22 coating, cast C22, TC4, and ND in 50 wt.% H$_2$SO$_4$ immersion testing at 50–70 °C for 72 h is illustrated in Figure 6. It is noted that the truncation and scale change of axis are adopted because of the large difference between the values of the four specimens.
As is clearly shown in Figure 6, the corrosion rate of the four specimens presents the same order from small to large at 50 °C, 60 °C, and 70 °C, which is C22 bulk < C22 coating < TC4 < ND. The corrosion rate curve of C22 bulk keeps close to zero through the entire testing, which indicates that corrosion barely occurs as the testing proceeds. Meanwhile, the curves of the C22 coating keep nearly level as that of C22 bulk when comparing them with those of TC4 and ND. The corrosion rate of TC4 and ND is drastically higher than that of C22 bulk and the C22 coating, i.e., approximately 20 times and 160 times greater than that of the C22 coating, respectively. It could be simply concluded that the corrosion resistance of the four specimens presents in a descending order as C22 bulk > C22 coating > TC4 > ND. More precisely, the C22 coating presents an almost similarly excellent and stable corrosion resistance as that of C22 bulk. The corrosion resistance of TC4 is poorer, and ND showed the worst corrosion resistance. The slight weakness of the C22 coating is assumed to result from the dominate degradation effect of the high Fe content and black micropores mentioned above.

The corrosion rate of C22 bulk, TC4, and ND increases with the increase in temperature, indicating the aggravation of corrosion caused by the temperature increase. In terms of testing time effect, the corrosion rate curves of both C22 bulk and the C22 coating keep approximately horizontal. This implies that the initial oxidation film of C22 bulk and the C22 coating has excellent corrosion resistance to efficiently prevent severe damage, the further diffusion of the corrosion medium, and the charge transportation [35–39]. In contrast, the corrosion rate of TC4 and ND reduces as the testing time increases, and the extent of the reduction of ND is larger than that of TC4. This implies that the initial oxidation film of ND and TC4 was severely damaged at the beginning of testing, and then the formation of a passive film retarded the corrosion to a nearly constant rate as time increased [21,22,58]. Moreover, the corrosion rate curves of the C22 coating at 50 °C and 70 °C present a similar trend, but the one at 60 °C presents a slight fluctuation. The corrosion rate at 60 °C has a negative value during 24 h–60 h, and the value after 72 h is slightly smaller than that at 50 °C. It is assumed that the localized corrosion and adhering corrosion products result in the unusual fluctuation, which was observed in the research on Ni–Cr–Mo alloy corrosion [38,39]. Hence, the measurement and calculation of corrosion rate were misled by this highly localized deterioration and incomplete removal of corrosion products [70]. Specifically, the negative corrosion rate at 60 °C probably resulted from the additional corrosion products remaining after the cleaning process. This speculation is discussed in detail with respect to the corrosion morphologies in the following section.

3.4. Corrosion Morphology and Analysis

The corroded surfaces of the laser cladding C22 coating and the cast Hastelloy C22 bulk in 50 wt.% H2SO4 immersion testing at 50–70 °C for 72 h duration are shown in Figure 7. As mentioned above, C22 bulk was barely corroded at 50 °C and 60 °C, and a barely distinct morphology change was found. Hence, only corrosion morphology at 70 °C was adopted for contrasting.

As is obviously shown in Figure 7, the C22 coating exhibits a distinct intergranular corrosion behavior, and the eutectics in the grain boundary are severely corroded with a bright corrosion product forming in the center. A similar intergranular corrosion occurs on C22 bulk, but a lower degree of corrosion is shown by the discontinuous defects in the grain boundary. Meanwhile, both the primary grains of the C22 coating and C22 bulk remain seemingly intact with a protective dense passive film forming on the surfaces. When comparing the corrosion morphologies of the C22 coating at different testing temperatures (Figure 7a–c), we observe that the material loss of eutectics is significantly aggravated as the temperature increases. In particular, the eutectics in the grain boundary of the C22 coating almost completely disappears at 70 °C, exposing several layers of primary grains deep in the interior. However, there is no significant difference between 50 °C and 60 °C, only slightly less bright corrosion products remaining at 60 °C. Moreover, the material loss mainly occurs in the black micropores and the interface between the prime grains and the eutectics, as marked by the arrows in Figure 7a,b. It is speculated that the corrosion occurs preferentially at these two locations, and the eutectics react synchronously with sulfuric acid to form dense corrosion products. In addition,
the material loss and dissolution of the corrosion products are aggravated as the temperature increases. The seemingly dense corrosion products remaining in the grain boundary are considered as the major factor that misleads the calculation of the C22 coating’s corrosion rate at 60 °C.

The elemental content analysis of the C22 coating corroded at 60 °C is shown in Figure 8. In contrast with the EDS analysis of the uncorroded C22 coating in Figure 5, the main elemental content of the primary grain (Point 1) barely changed, but there are obvious differences between the remaining bright corrosion product (Point 2) and uncorroded eutectics. After the increase of elemental oxygen, the bright corrosion product contains significantly more elemental chromium and slightly less elemental molybdenum. Combined with the observation about corrosion morphology, it is concluded that the more severe intergranular corrosion is the dominant corrosion behavior of the C22 coating, comparing to that of C22 bulk. The molybdenum-depleted region adjacent to the molybdenum-segregated region in the eutectic is preferentially attacked, resulting in the primary grain–eutectic bond weakening and leading to the intergranular corrosion [36,40,71]. Simultaneously, the formation of chromium containing corrosion products causes the chromium depletion in the eutectic, and further degrades the corrosion resistance [48,49,69]. In addition, a potential difference exists between the prime grains and the eutectics because of the compositional heterogeneity, which establishes micro-batteries between the prime grains and the eutectics with smaller grain-sized anodes. These formed smaller anode micro-batteries establish a micro-scale corrosion cell system and accelerate the intergranular corrosion [50,66]. Both the corrosion reaction and the electrochemical reaction are to a certain extent controlled by the ion diffusion process in the testing solution; therefore, the corrosion rate of the C22 coating increases with the increase in temperature [36,38,72]. The synergistic effect of a high Fe content and black micropores resulting from the laser cladding process is considered as the primary cause of the degraded corrosion resistance of the C22 coating compared with that of C22 bulk.

Figure 7. Surface morphologies of (a–c) the C22 coating corroded at 50–70 °C and (d) C22 bulk corroded at 70 °C: (a) 50 °C, (b) 60 °C, and (c) 70 °C.
4. Conclusions

1. A Hastelloy C22 coating fabricated by coaxial laser cladding technology is composed of a $\gamma$-Ni solid solution and carbide Ni$_6$Mo$_6$C$_{1.06}$, essentially similar to the cast Hastelloy C22 bulk.

2. The microstructure of the C22 coating mainly contains eutectic and dendrite, and the distribution of solidification from the bottom to the top is cellular, columnar, and equiaxed. These characteristics are similar to Ni–Cr–Mo coatings fabricated by powder presetting laser cladding.

3. The corrosion resistance of the C22 coating in a simulated sulfuric acid dew point corrosion environment is very close to that of C22 bulk, and approximate 20 times that of titanium alloy TC4 and 160 times that of corrosion-resistant steel ND. The increase in temperature obviously aggravates the corrosion of the four specimens.

4. The corrosion behavior of the C22 coating is intergranular corrosion. The segregation of molybdenum, chromium containing corrosion products, and smaller anode micro-batteries leads to the more severe corrosion compared to that of C22 bulk. The ion diffusion process is the dominant influence factor of the corrosion rate.

5. In consideration of the similar performance and the lower cost compared to those of a whole C22 alloy application, the laser cladding C22 coating is a potential technology for preventing sulfuric acid dew point corrosion in low-temperature devices in coal-fired boilers, such as FDG systems and backend ductworks.

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