Analysis on the localized corrosion of hydraulic support after short-term service in coal

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Abstract

The corrosion of hydraulic support has brought huge economic losses to coal enterprises. Using laser cladding stainless steel coating on the surface can improve its wear resistance and corrosion resistance. However, dense corrosion pits appeared on the hydraulic support coated 304 stainless steel after 25 d use in a coal mine. At present, there are few studies on the corrosion effect of this laser cladding layer under the actual mine. In this work, the oxide scales are characterized by 3D Optical Microscope (OM), Scanning Electron Microscopy (SEM) equipped with x-ray Energy Spectrometer (EDS), x-ray Photoelectron Spectrometer (XPS) and x-ray Diffraction (XRD) techniques. Based on the results, the localized corrosion mechanism is discussed. It was found that the corrosion products were mainly a mixture of (Fe,Cr)2O3 and CaCO3. The maximum pitting rate calculated from the depth data of pits, which were collected by ultra-depth of field optical microscopy, was 2.32 mm a−1. The mechanism of corrosion of hydraulic support was discussed form perspectives of the under-deposit corrosion and corrosive ions (i.e. Cl− and S2−) in the environment.

1. Introduction

Safety and efficiency are the key issues in coal mine development. Hydraulic support plays the role of supporting the pressure of coal seam, which is a main support equipment of coal mining system and the basic structure to ensure the safety of coal mining [1, 2]. However, the underground environment of coal mines contains a large number of corrosive media, such as Cl−, CO2, SO2, H2S, etc, and a high relative humidity (more than 75%). The hydraulic support may suffer from severe corrosion, and the localized corrosion pits will deteriorate its tightness and then reduce the oil pressure and bearing capacity. For this reason, the hydraulic support of many mines will be out of the well for overhaul after one year [3, 4]. Therefore, prolonging service life of support system through relevant technologies is of great significance to reduce the maintenance cost of coal mine [5, 6]. Electroplating is one of the common anti-corrosion technologies used in coal mine [7]. However, at present, it is advanced and environment-friendly to use laser cladding technology to coat stainless steel on the surface of the support. Compared with electroplating, the coating prepared by laser cladding is metallurgically combined with the substrate, so it has good adhesion, wear resistance, corrosion resistance and service life 5 ~ 6 times that of traditional hydraulic supports. Xu et al [8] produced 304 stainless steel coating on the surface of 45 steel using laser cladding method, significantly improving its wear resistance and corrosion resistance. Therefore, the application of laser cladding stainless steel coating can reduce the maintenance frequency of the support, improve the production efficiency and protect the environment, which is of broad and far-reaching significance for building an energy-saving society.

A dense passive film can form on the surface of stainless steel in atmospheric environment, which makes its excellent corrosion resistance [9]. However, the presence of corrosive ions such as Cl− and S2− may induce the severe localized corrosion [10]. Localized corrosion is due to local breakdown of the passive film [11]. Various
theories have been proposed for Cl− could induce breakdown of the passive film. Ion penetration theory shows that Cl− incorporates into and migrates through the passive film, resulting in film breakdown when reaching upon the metal/passive film interface [12]. Competitive ion adsorption theory suggests Cl− adsorption is favoring over O2− adsorption when O2− and Cl− anions are adsorbed onto metal surfaces at the same time. Thus, passive film is easily broken down [12]. Complex ion formation theory suggests that Cl− ions are adsorbed around the cations on the surface of passive film to form complexes. The complex is highly soluble, resulting in local thinning of the passive film and further forming a complex with Cl−. The formation and dissolution of high energy complexes lead to film breakdown [13].

The effect of sulfide on deterioration of corrosion resistance of stainless steels and low alloy steels has been investigated previously [14]. Sulfide mainly exists in the outer layer of stainless steels passive film in sulfide-containing environments, which is composed of Cr(OH)3, FeS2 and FeS. This result in thinner barrier layer, lower charge transfer resistance and weaker protection of the passive film on the substrate [15]. Once sulfide contacts with acidic medium, it is easy to convert to hydrogen sulfide [16]. Ding [17] finds that the presence of H2S in solutions increases the current density of cathodic and anodic, and then increasing the corrosion susceptibility of 316 L. Wang [18] points out that the addition of H2S increases the density of acceptor and donor, leading to the degradation of passive film [19].

The corrosion of hydraulic support in mine belongs to atmospheric corrosion. Generally, the nature atmospheric corrosion test of stainless steels will almost take several years, which cannot meet the timely needs of research [20]. Therefore, it is necessary to use accelerated corrosion methods, such as cyclic corrosion tests, that is, cyclic corrosion of metal surface during humidity and drying [21]. The results show that several small pits appear on the surface of 316 L stainless steel after 10 d of accelerated cyclic corrosion in the presence of chloride ions in the atmosphere. And a large area of corrosion products appeared after 60 d of corrosion [20]. In this study, the corrosion part is selected from hydraulic support with laser cladding 304 stainless steel. Local corrosion occurred after 25 d of actual use under the coal mines in Northern Shaanxi, China. It is indicated that the corrosion rate in this case is higher than that in traditional atmospheric corrosion conditions without accelerated cyclic corrosion, and the local corrosion mechanism of these need to be clarified.

Since the corrosion process is predominantly controlled by electrochemical reactions. Many researchers have been used electrochemical measurements to monitor of atmospheric corrosion instantaneously. Kelvin probes have been used by capture the electrochemical signal to map surface Volta potentials under atmospheric corrosion conditions [22]. However, this method cannot be proper for actual field monitoring. EIS technique has great advantage as a non-destructive procedure for the study of atmospheric corrosion and has successfully been used to study the effect of thin electrolyte layers. Nishikata [23] et al reported on the atmospheric corrosion of steels under wet and dry cyclic conditions using EIS in laboratory simulated as well as field [24] environments. Dubuisson [25] et al studied the atmospheric corrosion of galvanised steel in an electrolytic droplet using EIS method. Nevertheless, in terms of the applicable atmospheric conditions required for accurate and reproducible EIS data acquisition as well as the analysis of EIS spectra under different atmospheric conditions, some questions remain unanswered. Additionally, the quantified parameters for monitoring the corrosion process are difficult to be satisfactorily discussed [26]. So, in this paper, the main monitoring techniques we choose are based on measurement of physical quantities (3D Optical Microscope, Scanning Electron Microscopy equipped with x-ray Energy Spectrometer, x-ray Photoelectron Spectrometer and x-ray Diffraction techniques), because that atmospheric corrosion in real mine is complex which is difficult to analysis. Based on the results, the local corrosion mechanism was discussed.

2. Experiment

2.1. Samples preparation

Type hydraulic support coated 304 stainless steel sampling had been kept in service for 25 d in the actual service process under the mine. Small coupons were cut from the corrosion position. Some coupons were inlaid with resin, carefully mechanically ground progressively with fine grit SiC paper up to 2000 grit, and finally mechanically polished with 1 μm diamond pastes to get the cross section. Other coupons were used for the follow-up analysis. In order to measure the depth of corrosion pit by 3D optical microscope, the corrosion products on the sample surface need to be removed. The corrosion products could be removed by ultrasonic cleaning the sample in 60 degree film remover for 20 min. The film remover is nitric acid (HNO3, ρ = 1.42 g ml−1) solution with a volume ratio of 1:10 to distilled water. In order to measure the depth of corrosion pit by three-dimensional scene, it was necessary to remove the corrosion products on the sample surface with film remover solvent. The formula of the membrane removal solvent is 10 ml nitric acid (HNO3, ρ = 1.42 g ml−1), add distilled water to prepare 100 ml solution. The method was to put the sample in film remover solvent at 60 °C for 20 min with ultrasonic. Other coupons were used for the follow-up analysis.
2.2. Characterization of the corrosion products

The micro morphology (including surface morphology and cross-section morphology) of the corrosion products was observed by TESCAN VEGA-III SEM under an acceleration voltage of 25 kV. The chemical composition was analyzed by EDS. The phase structure was identified by PW1700 x-ray diffractometer. The x-ray photoelectron spectroscopy was collected by an x-ray photoelectron spectrometer (XPS, ESCALAB 250) with Al Kα x-ray excitation source, under the target voltage of 15 kV and the power of 150 W.

2.3. Corrosion rate evaluation

After removing the surface corrosion products, the corrosion pit was measured by ZEISS Smarzoom5 3D optical microscope. The 3D profile was collected and the pit depth was measured. The maximum pitting rate (MPR) was calculated according to equation (1), where t represents the corrosion duration (day), and d represents the maximum pitting depth (mm).

\[
M P R = \frac{d}{t} \times 365
\]

2.4. Chemical compositions of the water and raw coal powder of coal mine

The chemical compositions of the underground water is shown in table 1, and the compositions of the raw coal powder is shown in table 2.
3. Results and discussion

3.1. Macro morphology and characteristics of corrosion

The macro corrosion morphology of hydraulic support coated 304 stainless steel in mine is shown in the figure 1(a). Except for local corrosion pits, most of the surface is still bright. Figures 1(b), (c) shows the...
morphology of corrosion products by the optical microscope. It is found that the damaged surface morphology is corrosion pits of different sizes containing black and red products.

The figure 2 shows the cross-section of the laser cladding coating, in which it can be seen that the cladding layer has good metallurgical combination with the matrix. The cladding layer is mainly composed of \( \alpha' \) martensite phase and retained austenite phase. In the microstructure observation, it is found that there are a large number of cellular crystals and equiaxed crystals in the upper part of the cladding layer, while the lower layer in contact between the matrix and the cladding layer is mainly long strip dendrites, which is mainly due to the heat dissipation through the matrix during the cladding process and the large temperature gradient, This kind of planar crystal structure [27] appears. The microstructure of the out coating is more uniform, so it has excellent corrosion resistance [28].

3.2. Analysis of the corrosion products

Figure 3(a) presents a complete surface morphology of the corrosion products on the hydraulic support coated 304 stainless steel. The surface is uneven and some parts of the oxide scale are exfoliated. Figure (b) shows the unpeeled bright area oxide scale in figure 3, and some tiny cracks exist in the bright area oxide scale. Figure 3(c) is high-magnification images of the oxide scales on the hole. It is also found that particles floating in the air adhere to the surface which is shown in figure 3(d). All EDS results of different positions are shown in table 3. EDS results indicate that the bright area oxide scale are mixture of iron oxide and calcium oxide (or calcium carbonate) with little sulfur compound. The thin oxide scales with small round particles in the hole area are iron oxides with enriched Cr. The adhered particles may be coal powder which contains calcium oxide or calcium carbonate.

Table 3. Chemical compositions on different regions detected by EDS (at%).

| Location | O       | Fe      | Cr  | Ca  | S  | C    |
|----------|---------|---------|-----|-----|----|-----|
| 1        | 34.47   | 34.86   | 26.68 | —   | 1.53 | —   |
| 2        | 72.83   | 3.31    | 1.23 | 9.54 | 0.45 | 10.54 |
| 3        | 69.59   | 2.30    | 0.40 | 8.80 | 0.24 | 15.90 |
| 4        | 57.07   | 3.82    | 1.18 | 36.54 | —   | 1.39 |

Figure 4 shows the cross-section morphologies of the oxide scales on the no spalling surface area. This oxide film contains numerous pores and voids. The EDS line scan results are shown in figure 5. It is found that the oxide layer is rich in Ca and small in S. However, stainless steel coating does not contain these two elements. The content of C in the outermost layer is highest which is possibly because the presence of resin or CaCO\(_3\). In order to analyze the composition distribution more accurately, EDS points distributed longitudinally of the oxide scale is shown in table 4. It can be seen that the outermost oxide layer is mixture of iron oxide with enrich Cr and CaCO\(_3\) (or CaO). While S is present in the inner layer, for which S may come from the adhered coal to the hydraulic support surface or H\(_2\)S in the atmosphere dissolving in the water film, which should lead to aggravate the corrosion rate [29]. The structure of the outer oxide scale which has a small amount of Ca\(^{2+}\) was loose and porous. This implies that the presence of Ca\(^{2+}\) could cause looseness of the scale structure. Zhao et al [30] found that the presence of Ca\(^{2+}\) made the formed oxide layer loose and unprotected, and pitting corrosion was more likely to occur. Therefore, the presence of calcium carbonate has an important effect on corrosion of steel.
In order to further analyze the chemical composition of outer layer of the oxide scale, XPS experiments were carried out. The peak splitting curve of O1s is shown in figure 6, which shows three peaks, namely Fe$_2$O$_3$ (529.98 eV), CaCO$_3$ (531.70 eV) and –OH (533.89 eV). The –OH in XPS is mainly the peak separated from H$_2$O [31, 32]. XPS results show that the oxide scales contain CaCO$_3$ rather than CaO.

Figure 7 shows the x-ray diffraction analysis which can judge the phase structure of the corrosion product. Combined with the morphology, EDS and XPS of the corrosion product, it can be judged from the XRD that the main component of the outer layer of corrosion products are mixture of Fe$_2$O$_3$ and CaCO$_3$.

In summary, the oxide layer formed on the surface of stainless steel after 25 d service in the atmospheric environment of mine is mixture of iron oxide with enrich Cr, sulfide and CaCO$_3$.

Table 4. Chemical compositions on different regions in the cross-section detected by EDS (at%).

| Location | O  | Fe  | Cr  | Ca  | S   | C   |
|----------|----|-----|-----|-----|-----|-----|
| 1        | 59.87 | 4.71  | 1.83 | 0.50 | —   | 32.49 |
| 2        | 75.70 | 11.61 | 4.54 | 1.21 | 0.64 | 4.33 |
| 3        | 76.10 | 11.10 | 4.46 | 1.10 | 0.64 | 4.31 |
| 4        | 76.30 | 13.97 | 3.55 | 0.87 | 0.69 | 2.55 |

Figure 5. The EDS line scan results along the cross section of the oxide scale.
3.3. 3D Analysis of corrosion pits

There are numerous individual pits formed which scatter across the hydraulic support surface. Figure 8 shows the 3D morphology and depth of pits. In many cases, the clear 3D visualization characterization can show enough information about the surface microtopography to support the analysis the size and shape of pits. From figure 8(c), it shows that some small pits merged large pits with other pits. This means that not all pits initiated at exactly the same time, and their growth rate was most likely different [33].

After the test for 25 d, the maximum depth of the pits measured according to the 3D microscopy is 159 μm, and the corresponding maximum pitting rate is 2.32 mm a⁻¹.

3.4. Analysis of the causes of corrosion

The results of SEM, EDS and XPS show that the corrosion products at the pitting are mainly a mixture of CaCO₃ and (Fe, Cr)₂O₃ and a small amount of sulfur-containing compounds. There are two main sources of CaCO₃: one is that there are more coal powder containing CaO floating in the mine, so they are easy to adsorb and deposit on the surface of the hydraulic support column. When the coal powder contacts with the condensed water solution, CaCO₃ will be generated. The second is that Ca²⁺ compounds are detected in the mine water sample. With the evaporation and adsorption of water vapor to the column surface, CaCO₃ in the liquid film will precipitate and deposit on the column surface. CaCO₃ covers the surface and forms a deposit layer. These two CaCO₃ deposits cause under deposit corrosion (UDC), which is easy to initiate pitting corrosion under the scale.
UDC is one of the most destructive forms of local corrosion. It produces uneven corrosion pits on the metal surface, sometimes several pits are connected together. However, the corrosion range is large or small, and the corrosion depth is different. Under the influence of gravity, the upward surface generally corrodes heavily, and 3 mm thick steel plate can be perforated within 3 ~ 24 months \([34, 35]\). This result indicates that the existence of the CaCO₃ deposit could reduce the protective performance of the stainless steel, allowing further corrosion of

![Figure 8](image_url1)

**Figure 8.** The 3D morphology and depth measurement of pits (a) depth of 159 \(\mu\)m; (b) depth of 114 \(\mu\)m; (c) depth of 102 \(\mu\)m; (d) depth of 132 \(\mu\)m.

![Figure 9](image_url2)

**Figure 9.** Schematic diagram of localized corrosion happened on the surface of hydraulic support, M represents iron or chromium here.
the material matrix. The main driving force of UDC is the chemical and physical differences between covered and uncovered areas. These differences may be differences in pH, or higher or lower oxygen concentrations, or sometimes a combination of all these factors [36]. Figure 9 shows the schematic diagram of under-deposit corrosion.

The nature of under deposit corrosion is electrochemical corrosion [37]. The general corrosion reaction of stainless steel is:

Cathodic reaction:

\[ 2H^+ + 2e^- \rightarrow H_2 \]  
(2)  

\[ O_2 + 2H_2O + 4e^- \rightarrow 4OH^- \]  
(3)

Anode reaction:

\[ Fe \rightarrow Fe^{2+} + 2e^- \]  
(4)  

\[ Cr \rightarrow Cr^{3+} + 3e^- \]  
(5)

Hydrolysis reaction of metal ions:

\[ Fe^{2+} + 2H_2O \rightarrow Fe(OH)_2 + 2H^+ \]  
(6)  

\[ Cr^{3+} + 3H_2O \rightarrow Cr(OH)_3 + 3H^+ \]  
(7)

Reaction of hydroxide with O2:

\[ 4Fe(OH)_2 + O_2 \rightarrow 2Fe_2O_3 + 4H_2O \]  
(8)

Dehydration reaction:

\[ 2Cr(OH)_3 \rightarrow Cr_2O_3 + 3H_2O \]  
(9)

It can be seen from the above reaction formula that the anode and cathode of electrochemical reaction correspond to oxygen poor area and oxygen rich area respectively which is shown in figure 9(a) [38]. In the cathode region, O2 obtains electrons to generate OH\(^-\) through the reduction reaction and the alkalinity of the solution in the cathode region is enhanced. The electronics can be supplemented from the anode. In the anode region, it can be seen that metal lose electrons to generate metal ions from the above reaction formula (4), (5) shown in figure 9(b). Because the metal matrix in the anode area is tightly covered by CaCO3 and other scale layers, O2 cannot enter the closed area under the scale to react with the Fe\(^{2+}\) or Cr\(^{3+}\) generated by the anode, which also leads to the enrichment of Fe\(^{2+}\) or Cr\(^{3+}\) in the anode area. Fe\(^{2+}\) or Cr\(^{3+}\) will further to react with H2O to form Fe(OH)\(_2\), or Cr(OH)\(_3\), and H\(^+\), resulting in the enhancement of the acidity of the solution under the scale and the continuous dissolution of the metal under the scale. Water comes from the water film formed by water vapor absorbed by the deposit layer which contains CaCO3, Cl\(^-\) and S\(^2-\) can pass through the scale layer to form HCl and H\(_2\)S with H\(^+\) for charge neutralization to maintain charge balance because Cl\(^-\) and S\(^2-\) have strong penetration [8]. They could initiate pitting corrosion of stainless steel in this high acidity environment under the scale, although the chromium content is high shown in figure 9(c). More and more Fe (OH)\(_2\) is generated under the solid deposit in the anode region with time, even mixed with the deposit which contains CaCO3. Fe(OH)\(_2\) is unstable which is easy to react with O\(_2\) and would form Fe\(_2\)O\(_3\) at the outer deposit layer. So the main composition of the outer surface scale is mixture of Fe\(_2\)O\(_3\) + CaCO\(_3\). The Fe\(^{2+}\) with positive charge accumulating in the anode area will attract Cl\(^-\) or S\(^2-\) to form FeCl\(_2\) or FeS. These generated precipitates of FeS and Fe\(_2\)O\(_3\) cannot be smoothly discharged from small cracks or pinhole openings, and gradually fill in the corrosion pit [39]. When the mixed oxide deposition is enough, spalling will occur shown in figure 9(d).

Therefore, when the metal surface is tightly covered by scale layer, it is difficult for stainless steel with high Cr content to form dense Cr\(_2\)O\(_3\) protective film. It is observed that once corrosion develops, corrosion pits increase and corrosion products increase. In CO\(_2\) corrosion of mild steel, the dense and adhesive FeCO\(_3\) layer formed on the steel surface has a protective effect, because this layer prevents the transportation of corrosive species toward the steel surface and blocks portions of the steel surface making them unavailable for corrosion [40]. When Ca\(^{2+}\) appearing, some studies claiming the corrosion rate is been decreased and the presence of Ca\(^{2+}\) could postpone the initiation of the pitting [41]. Conversely, some researchers claim the opposite conclusions. The corrosion rate increasing with an increase in the Ca\(^{2+}\) concentration [42]. Higher concentrations of Ca\(^{2+}\) could leads to the change of corrosion products from protective FeCO\(_3\) to unprotected calcium carbonate and the corrosion rate increases with time [43]. This confusion found in the public literature is because the pH value of the aqueous solution is not measured or reported. Only higher pH value is associated with the formation of protective FeCO\(_3\) layer [40]. Therefore, any effect of Ca\(^{2+}\) concentration must be analyzed in combination with the overall hydrochemical effect. As discussed earlier, the pH value under deposit is low, and the CaCO\(_3\) layer is porous, as shown in figure 4, which could not protect the substrate. Therefore, the role of CaCO\(_3\) in this paper is mainly to cause under deposit corrosion rather than protection.
4. Conclusion

The case of localized corrosion of laser cladding 304 stainless steel coated hydraulic support after short-term service was studied in the present work by 3D Optical Microscope, SEM, EDS, XPS and XRD techniques. Based on the results, following conclusions can be made.

(1) For the localized corrosion region, the porous outer surface is a mixture of CaCO₃ and Fe₂O₃, where the compact inner layer is mainly composed of Fe₂O₃ enriched Cr and S.

(2) The deposit of CaCO₃ on the surface of the hydraulic support may induce the initiation of pitting corrosion, the maximum pitting rate measured by 3D morphology is 2.32 mm a⁻¹.

(3) The localized corrosion of hydraulic support is mainly ascribed to the under-deposit corrosion induced by the deposit of CaCO₃ and Fe₂O₃, as well as the corrosive ions of Cl⁻ and S²⁻.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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