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Studies about aging and corrosion of galvanized steel and polyvinyl chloride shielded cable caused by fireproof mud

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Abstract

Fireproof mud is a widely-used sealing and plugging material in power and construction industry, yet it may cause aging/corrosion problems to its directly contacted materials, leading to high safety risk. However, studies about the aging/corrosion behaviours caused by fireproof mud are surprisingly rare. To fill in such a research gap, aging and corrosion of galvanized steel and polyvinyl chloride (PVC)-shielded cable caused by fireproof mud under simulated hygrothermal environment have been systematically investigated, based on which the mechanisms of these processes have been proposed. The results show that the corrosion of galvanized steel is mainly influenced by chloride anions from the fireproof mud, which can be aggravated/alleviated if the fireproof mud is acidic/alkaline. For the aging of PVC shielded cable, one main reason is acidic/alkaline chemical corrosion caused by acidic/alkaline fireproof mud, the other is radical chain reaction of the PVC polymer in hygrothermal environment. These discoveries can offer important guidance for correct and safe use of fireproof mud in industries, thus ensure safe industrial production.

1. Introduction

The research about aging and corrosoing of galvanized steel and cable is of great significance due to their fundamental role in power industry and construction industry. So far, aging and corrosion of galvanized steel and cable in atmosphere [1–4], soil [5–8], seawater [9–11] and many other types of environment [12–15] have been thoroughly studied, and the results demonstrate that the corrosion of steel and cable is affected by many factors, such as temperature, pH, humidity and dusts/active anions/molecules in the environment and so on [16–20]. For example, chloride can induce the corrosion of steel and an increasing concentration of chloride could reduce the corrosion resistance of steel [17]. Quennehnen et al has explored aging mechanism of PVC shielded cables, showing that oxidation, effect of water ingress, loss or migration of plasticizers and dehydrochlorization may be responsible for the aging and reduced electric resistance of the cables [20]. These researches have laid primary foundations for further investigations about the aging/corrosion behaviours and mechanisms of metals and cables under some rare environment.

Fireproof mud is soft flame resistant material with appearance of muddy and plastic solid. According to its constitues, fireproof mud can be roughly divided into three catalogues: siliceous acidic one, magnesium-calcia alkaline one and neutral one that contains both siliceous and magnesia-calcia constituents. Due to its very high deformability and flame-retarded ability, fireproof mud is widely used as sealing and plugging material in power plant, transformer substation and mining enterprises. However, investigations about some accidents in recent years find that direct contact of fireproof mud with metals/cables may cause their aging and corrosion behaviours, leading to gas leaking, short circuit problems etc. For example, corrosion of shielded wire of signal cable in terminal box can cause data corruption or even crash of monitoring system. Besides, the operational stability of electric power equipment is also severely threatened by corrosion of switch, bare ground wires and
other similar parts. Therefore, to take effective preventions, it is of great importance to understand the aging and corrosion behaviour caused by fireproof mud. Surprisingly, so far there have been hardly any reports of this research project, making the filling of such a research gap urgent and vital.

With the above considerations, representative types of fireproof mud in power and construction industry were selected and their influences on aging and corrosion of galvanized steel and polyvinyl chloride (PVC) insulated cable were studied, based on which mechanisms of aging and corrosion caused by fireproof mud have been suggested. To the best of our knowledge, our work about aging and corrosion resulted from fireproof mud is pioneering and can provide reliable insights for correct and safe use of fireproof mud in industrial constructions.

2. Experiments

2.1. Materials

The representative types of fireproof mud used in power and construction industry have product model as follows: DR-A3-CS02, DR-A2-AB-2, DR-A3-XA09, DR-A3-JH-2, DR-A3-CD02 and DR-A3-PC-YJDL, which were labelled as F1, F2, F3, F4, F5 and F6, respectively. Galvanized steel is produced by Kunshan Tengda Metal Products Limited Company, which was cut into slices with sizes of 30 mm × 30 mm × 1 mm in experiments. The cable explored in this work is insulation wire with copper core and PVC outer layer, which was cut into several segments with length of 2 cm.

2.2. Methods

2.2.1. Accelerated aging and corrosion in simulated hygrothermal environment

Each type of fireproof mud (from F1 to F6) was separately pasted on top surface of different galvanized steel slices and then left in climate chamber for 30 days (figure S1 (available online at stacks.iop.org/MRX/8/046523/mmedia)), of which the temperature and humidity was 45 °C and 90%, respectively. The as-obtained samples were labelled as S1, S2, S3, S4, S5 and S6, respectively. In control experiment, galvanized steel slice without any pasted fireproof mud (labelled as S0) was simultaneously left in the same climate chamber. In addition, an original galvanized steel slice with no aging or corrosion experiments was labeled as S.

Each type of fireproof mud (from F1 to F6) was separately pasted on surface of different PVC cable segments and then left in climate chamber for 90 days (figure S2), of which the temperature and humidity was 45 °C and
The as-obtained samples were labelled as C1, C2, C3, C4, C5 and C6, respectively. In control experiment, PVC cable segment without any pasted fireproof mud (labelled as C0) was simultaneously left in the same climate chamber. In addition, an original PVC cable segment with no aging or corrosion experiments was labeled as C.

2.2.2. Electrochemical measurements
Linear sweep voltammetry (LSV) tests were performed on a CHI660E electrochemical workstation with a scanning rate of 3 mV s$^{-1}$ to investigate the corrosion behaviour of the galvanized steel. A three-electrode system was applied with Pt, saturated calomel electrode and galvanized steel (10 mm $\times$ 10 mm $\times$ 1mm) as auxiliary electrode, reference electrode and working electrode, respectively. The working electrode and the auxiliary electrode were separated by porous ceramic membrane. NaCl solution was used as electrolyte.

2.3. Characterizations
X-ray diffraction (XRD) and scanning electron microscope (SEM) were utilized to characterize the constitutes and morphologies of samples. XRD was performed on X’Pert PRO (PANalytical Netherlands) using Cu target as x-ray source with a step length of 0.04, of which K$_{\alpha}$, tube current and tube potential were 0.154056 nm, 40 mA and 40 kV, respectively. SEM was carried out with a field emission scanning electron microscope (S-4700 II Hitachi, Japan). Energy dispersive spectrometer (EDS) was carried out with energy dispersive spectrometer (Thermo NORAN, USA). FT-IR was performed on fourier transform infrared spectrometer (Thermo Nicolet 6700, USA).

3. Results and discussions
3.1. Main constitutes of fireproof mud
To study the constitutes of the fireproof mud, XRD tests have been performed. The results (figure 1) show that the main inorganic constitute for fireproof mud F1, F2, F5 and F6 is acidic with SiO$_2$. While for fireproof mud F3 and F4, its main inorganic constitute is alkaline CaMg(CO$_3$)$_2$. The pH tests of the fireproof muds dispersions, which were prepared by dissloving 1g of each fireproof mud in 30 ml of deionized H$_2$O under ultrasonication for 60 min at room temperature, provide information consistent with the XRD results. The pH value for F1, F2, F3, F4, F5 and F6 was 5.2, 4.8, 7.9, 8.3, 5.7 and 6.0, respectively, showing different acidiness/basicity of the fireproof muds. In addition, there are some minor additives in fireproof mud, such as kaolin, bentonite and Al(OH)$_3$ etc. Among these minor constitutes, kaolin can increase refractoriness and plasticity of fireproof mud, bentonite can enhance its adhesiveness and plasticity, and Al(OH)$_3$, as one of the most widely applied inorganic fire retardant, can prevent smoking and producing toxic gases [21]:

$$
2\text{Al(OH)}_3(s) \xrightarrow{180 - 200 \degree C} \text{Al}_2\text{O}_3(s) + 3\text{H}_2\text{O(g)}
$$

(1)
3.2. Morphology of galvanized steel corroded by fireproof mud

To further investigate micro morphology of the galvanized steel, SEM tests of samples S0-S6 have been done. Figure 2 shows that, after left in climate chamber for 30 days, all the samples were corroded but with different corrosion degrees. Sample S0 was just slightly corroded at its original scratches (figure S3), which was due to the destroy of passive film at the scratch, causing adsorption of some active anions functioning as anode (the surrounding metal surface with higher potentials function as cathode) and finally resulting in formation of pitting holes [22]. Compared with sample S0, sample S1-S6 showed more severe corrosion with large area of pitting holes and corrosion pits (figure 2), suggesting the paste of fireproof mud under hygrothermal environment would aggravate the corrosion of galvanized steel.

It’s worth noting that among S1–S6, the corrosion degree of S3 and S4 was relatively lighter than the other samples. For S1, S5 and S6, granular corrosion products were formed on their surfaces, while for S2, extensive cracked corrosion layers appeared. Since the fireproof mud pasted on S3 and S4 contains basic CaMg(CO3)2, while the fireproof mud pasted on S1, S2, S5 and S6 mainly contain acidic SiO2, it is suggested that the acidiness/basicity of fireproof mud have big influences on the corrosion of galvanized steel.

EDS analysis of the corroded area of each sample have been done (figure S4). The results (table 1) show that the corrosion products of these samples contain elements such as Fe, Zn, Al, Si, C, O, K, Ca, Mg and Cl. Among these elements, Al, Si and Cl are mainly from fireproof mud. For S0, EDS results showed that it was just slightly corroded with minor change of its Fe substrate, which is in accordance with the results of SEM analysis. On the contrast, for S1-S6, the content of elements C and O significantly increased compared to that of S, which may be resulted from both increase of corrosion products and directly-contacted fireproof muds. In addition, it should be noticed that the content of Cl in S1-S6 had remarkable increase compared to that of sample S0, indicating that the Cl might be a main reason leading to the corrosion of galvanized steel [23].

### Table 1. EDS results of corrosion products of sample S, S0 and S1-S6.

| Sample | C   | O   | Al  | Cl  | Si  | Fe  | Zn  | K   | Ca  | Mg  | S   |
|--------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| S      | 5.15| 2.58| 0.52| 0.11| —   | 3.38| —   | —   | —   | —   | —   |
| S0     | 5.25| 2.63| 1.29| 0.26| —   | 3.85| —   | —   | —   | —   | —   |
| S1     | 24.68| 13.71| 3.52| 5.91| 4.37| 2.97| 44.10| 0.24| 0.50| —   | —   |
| S2     | 29.76| 15.69| 2.28| 6.88| 3.45| 5.79| 34.74| 0.53| 0.58| 0.30| —   |
| S3     | 20.62| 14.27| 2.26| 4.63| 3.64| 3.50| 48.10| —   | 2.64| 0.34| —   |
| S4     | 15.00| 3.04| 2.03| 1.48| 0.65| 4.14| 71.43| —   | 1.95| 0.28| —   |
| S5     | 15.42| 20.47| 2.51| 1.27| 20.02| 3.70| 35.09| 1.21| 0.31| —   | —   |
| S6     | 16.10| 20.63| 0.80| 2.67| 6.56| 49.20| 2.95| —   | 0.40| 0.45| 0.11| 0.13|
3.3.3. Possible corrosion mechanism of galvanised steel caused by fireproof mud

Based on the above results and discussions, it is suggested that the corrosion process of galvanised steel caused by fireproof mud under simulated hygrothermal environment may include three stages. The first stage is active dissolution of the coating surface of galvanised steel. When corrosion initially occurs, the original scratches on the fireproof mud under simulated hygrothermal environment may include three stages. The first stage is active dissolution of the coating surface of galvanised steel. When corrosion initially occurs, the original scratches on
the surface of galvanized steel easily adsorb Cl\(^-\) from fireproof mud. Since Cl\(^-\) is a strong de-passivator with small radius, it can enter the Zn coating layer of galvanized steel, forming soluble salts with metallic cations and leading to locally corroded Zn coatings [23]. Related reaction equations are as follows:

\[ \text{anode: } Zn \rightarrow Zn^{2+} + 2e^-; \]
\[ \text{cathode: } O_2 + 2H_2O + 4e^- \rightarrow 4OH^- . \]

The Zn\(^{2+}\) produced in anode would move toward cathode and react with OH\(^-\):

\[ Zn^{2+} + 2OH^- \rightarrow Zn(OH)_2; \]
\[ Zn(OH)_2 \rightarrow ZnO + H_2O; \]
\[ 5Zn^{2+} + 2Cl^- + 8OH^- + H_2O \rightarrow Zn_3(OH)_8Cl_2 \cdot H_2O \]

At the second stage, as the Zn coating layer is locally corroded, the corrosion current density rapidly increases and the Cl\(^-\) anions may accumulate in corroded holes, leading to further development of the corrosion towards inner part of steel [24–26]. With the development of the corrosion, the Zn coating gets destroyed and partial Fe-substrate is exposed, resulting in the formation of Fe-Zn galvanic corrosion. Under this situation, the Zn coating functioning as sacrificial anode provides cathodic protection for the Fe-substrate. The erosion from Cl\(^-\) makes tight and insoluble zinc complexes on the surface of the Zn coating replaced by loose and soluble chloride compounds, which could significantly speed up the corrosion process of the Zn coating layer. According to researches of Keddam et al the main products of corroded Zn coating are tetrabasic zinc chloride, basic zinc carbonate and zinc oxide under environment with abundant Cl\(^-\) [27].

Last, as the Zn coating gets severely corroded and quickly depleted, large area of the Fe-substrate becomes uncovered and corroded, which can be greatly accelerated by the Cl\(^-\) in environment due to two reasons. One reason is that the invasion of Cl\(^-\) would weaken interactions between the Fe-substrate and the rust layer, causing loose or crack of the rust layer. As a result, the ability of the rust layer to inhibit dissolution and diffusion of the corrosion products is decreased. The other reason is that some corrosion product of the Fe-substrate, such as γ-FeOOH, is unstable with strong reducibility, which can increases active region of the cathodic reaction [26, 28, 29]. In addition, the presence of these corrosion products can benefit the diffusion of Cl\(^-\) in rust layer and thus facilitates the corrosion of the Fe-substrate [30, 31]. Typical reactions of this stage are as follows:

\[ \text{anode: } Fe \rightarrow Fe^{2+} + 2e^-; \]
\[ \text{cathode: } O_2 + 2H_2O + 4e^- \rightarrow 4OH^- . \]

The Fe\(^{2+}\) cations move toward cathodic region while OH\(^-\) and Cl\(^-\) move in an opposite direction. As a result, Fe(OH)_2 will form near the active region of anode:

\[ Fe^{2+} + 2OH^- \rightarrow Fe(OH)_2; \]
\[ Fe(OH)_2 + O_2 \rightarrow FeO_2, H_2O; \]
\[ Fe(OH)_2 + O_2 \rightarrow FeOOH. \]

The Fe(OH)_2 protection layer would then be damaged due to the invasion of Cl\(^-\):

\[ Fe(OH)_2 + Cl^- \rightarrow FeOH^+ + Cl^- + OH^- . \]

Furthermore, it needs to be pointed out that acidic environment is conducive to decreasing the corrosion resistance of galvanized steel. In experiments of electrochemical corrosion, H\(^+\) is reduced at the cathode. The lower the pH value, the stronger the depolarization of H\(^+\) to the cathode, resulting in an increase of the electric potential of the steel. When the pH value is low enough, the passive film of galvanized steel becomes corroded [32–34]. Meanwhile, H\(^+\) can inhibit the formation of passive film by dissolving metal oxides/hydroxides, which are the main compositions of passive film. These discoveries are in consistency with our results that sample S1, S2, S5 and S6 corroded by acidic fireproof mud showed more severe corrosion than that of other samples under the same environment.

3.4. Aging and corrosion of cable by fireproof mud

3.4.1. Morphology of cable corroded by fireproof mud

The outer layer of the cable studied in our experiments is mainly composed of polyvinyl chloride (PVC) with accessory ingredients such as plasticizer, antioxidant, colorant and so on [20]. C1-C6 pasted with fireproof mud and C0 without fireproof mud were put in the same climate chamber simultaneously. After 90 days, it is observed that the surface of C0 had no obvious change (figure S5). On the contrary, quite large dark spots appeared on the surface of C1, C2 and C3 (figure S6), and the dark round-shaped spots appeared on C2 even had clear cracks. For C4, C5 and C6, yellow spots (C4) and light yellow spots (C5 and C6) formed on their surfaces (figure S6). These results indicate that fireproof mud can aggravate aging of the cable. To further investigate the micromorphology of these samples, SEM tests have been performed (figure 4). Compared with the intact surface
of C0 (figure S5), obvious cracks appeared on the surfaces of C1-C6. In addition, large particles and loose pores can also be observed, confirming that fireproof mud can cause significant aging/corrosion to the cable.

3.4.2. FTIR spectra of cable corroded by fireproof mud
Figure 5 shows FTIR spectra of C0 and C1-C6. Peaks at 790 cm$^{-1}$ and 730 cm$^{-1}$ correspond to the stretching vibration mode of C-Cl and peaks at 1014 cm$^{-1}$ is characteristic of C-O stretching. Peaks in the range of 1460–1260 cm$^{-1}$ are due to deformation vibration of -CH$_2$ and peaks in the range of 3700–3300 cm$^{-1}$ are resulted from absorption of -OH. In addition, compared with the FTIR spectrum of C, an absorption peak of C=O appeared at 1720 cm$^{-1}$ in the FTIR spectra of C0 and C1-C6, indicating some oxidation reactions occurred at the surface of the cable, which might be a main path for the corrosion of cable [20].

3.4.3. Possible corrosion mechanism of cable caused by fireproof mud
Based on the above results and discussions, the corrosion of the cable may occur via oxidation of its PVC surface, which could cause destruction of the chain structure of PVC and then corrosion of the cable. For the occurrence of the oxidation reactions, there are two possible reasons [20]. One is that the fireproof mud itself contains some acidic or alkaline constituents, e.g., SiO$_2$ and CaMg(CO$_3$)$_2$ etc, which could cause acidic or alkali corrosion to the PVC surface of cables. The other is that the fireproof muds contain some intrinsic moisture (as demonstrated by measurement results of water content of the fireproof muds via weight-loss method, the intrinsic water content
of the fireproof muds was 9.76% for F1, 12.48% for F2, 5.15% for F3, 5.45% for F4, 8.16% for F5 and 5.82% for F6, and some minor additives of the fireproof mud, e.g. bentonite, can easily absorb water and ions from surrounding environment, leading to increase of the humidity of the PVC surface. The high humidity together with heat produced by the current when the cable works, would form hygrothermal environment around the surface of cable and then initiate radical chain reactions of PVC (equations (13)-(22)), resulting in aging and corrosion of the cable finally [35, 36]: 

(1) Chain initiation (PH: polymer, P.-polymer macromolecule free radical):

\[
\text{PH} \xrightarrow{\text{heat/light}} \text{P}^- + \text{H}^+; \quad (13)
\]

(2) Chain propagation:

\[
\begin{align*}
\text{P}^- + \text{O}_2 & \rightarrow \text{POO}^- , \quad (14) \\
\text{POO}^- + \text{PH} & \rightarrow \text{POOH} + \text{P}^-, \quad (15) \\
\text{POOH} \xrightarrow{\text{heat/light}} \text{PO}^- + \text{HO}^-, \quad (16) \\
2\text{POOH} & \xrightarrow{\text{heat/light}} \text{PO}^- + \text{POO}^- + \text{H}_2\text{O} \quad (17)
\end{align*}
\]

and

\[
\text{PO}^- + \text{PH} \rightarrow \text{POH} + \text{P}^-; \quad (18)
\]

(3) Chain termination:

\[
\begin{align*}
2\text{POO}^- & \rightarrow \text{POOP} + \text{O}_2, \quad (19) \\
2\text{POO}^- & \rightarrow \text{non} \rightarrow \text{reactive products}, \quad (20) \\
\text{P}^- + \text{POO}^- & \rightarrow \text{POOP} \quad (21)
\end{align*}
\]

and

\[
2\text{P}^- \rightarrow \text{P} - \text{P}. \quad (22)
\]

4. Conclusions

In summary, to fill in the research gap of aging and corrosion caused by fireproof mud to its directly contacted materials, representative types of fireproof mud in industry have been selected and their influences on aging and corrosion of galvanized steel and PVC insulated cable have been investigated, respectively. The results show that the chloride element from the fireproof mud may be the main reason for initiating and accelerating the corrosion of galvanized steel. Besides, the acidiness/alkalinity of fireproof mud can not only influence the corrosion process of galvanized steel, but also cause acidic/alkaline corrosion to the PVC surface of cable. Furthermore, some minor additives of the fireproof mud such as bentonite is conducive to formation of hygrothermal atmosphere on the PVC surface, which can facilitate the aging and corrosion of cable as well. Our research can offer scientific guidances to reasonal use of the fireproof mud in industry.

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