



## Full Length Article

# Corrosion behaviors of polysiloxane-ferroferic oxide coating coated on carbon steel in NaCl solution and geothermal water

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## ABSTRACT

Corrosion from plants and pipelines made of carbon steel is a severe problem for the utilization of geothermal energy. In order to control corrosion, various polysiloxane-ferroferic oxide composite coatings were successfully fabricated on carbon steel substrates. The morphologies and chemical compositions of these composite coatings were characterized. The thermal stability was tested by thermal gravity analysis (TGA). Wettability was investigated by contact angle measurement. Equivalent thickness was measured by weight method. The corrosion behaviors of composite coatings were tested by potentiodynamic polarization curves and electrochemical impedance spectroscopy (EIS) in 3.5 wt.% NaCl solution and simulated geothermal water of Huabei oilfield. According to potentiodynamic polarization curve and EIS, the inhibition of corrosion for each carbon steel coated with composite coating is improved, because the corrosion current density decreases by one or two orders of magnitude. Corrosion mechanism for carbon steel coated with composite coating was analyzed with EIS measurement and equivalent circuit simulation. TGA of various polysiloxane coatings shows less than 8% weight losses for all polysiloxane coatings tested at 300 °C. The composite polysiloxane coatings show good corrosion resistance and thermal stability.

## 1. Introduction

Geothermal energy with the characteristics of low cost, wide distribution, low pollution and renewability has drawn much attention (Cai et al., 2009; Liu and Zhu, 2011). Carbon steel is the main material used for the utilization of geothermal energy, which has the features of good machinability, low price and abundance. However, the corrosion of carbon steel is a severe problem (Bai et al., 1998). And many corrosion resistance methods have been suggested. In recent years, many new-style coatings for corrosion resistance have also been fabricated such as superhydrophobic coating (Ning et al., 2011; Yu et al., 2013a; de Leon et al., 2012; Hui Xu et al., 2011; Fan et al., 2014; Liu et al., 2014a; Zhang et al., 2014; Ishizaki and Sakamoto, 2011; Gao et al., 2014; Ishizaki et al., 2010; Liu et al., 2014b), cation-exchange pigment (Granizo et al., 2012), photoelectrochemical corrosion resistance coating (Sun et al., 2014; Ohko et al., 2002) and nanocontainer loaded with corrosion inhibitor (Borisova et al., 2011). While the traditional coatings used to protect carbon steel from corrosion are usually inorganic, organic or organic-inorganic hybrid coatings or films. These coatings can act as a kind of physical barriers which retard electrolyte

to penetrate into the surface of carbon steel.

Several techniques have been used to fabricate inorganic films on carbon steel. Magnetron co-sputtering method could be used to fabricate Zn-Mg corrosion resistance coating. In 3.5 wt.% NaCl solution, the coating could form a passive layer which can improve the corrosion resistance performance (Yao et al., 2013). Electroless Ni-Cu-P-ZrO<sub>2</sub> inorganic composite coatings could be served as corrosion resistance film through electroless plating technique. The incorporation of ZrO<sub>2</sub> in Ni-P coating showed better corrosion resistance than plain Ni-P coating and the introduction of Cu into Ni-P-ZrO<sub>2</sub> strengthened the protection ability against corrosion media (Ranganatha et al., 2012). The Sol-gel coating technique with the features of easy fabrication and low toxicity was also applied to prepare corrosion resistance coatings such as Y<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> film (Domínguez-Crespoa et al., 2009) and ZnO-TiO<sub>2</sub> coating (Yu et al., 2013b). Polyphosphate-based corrosion inhibitor was also used as corrosion resistance pigment coated on steel (Naderi and Attar, 2009).

Although inorganic corrosion resistance film mainly composed of metallic oxide, metal or insoluble inorganic salt presents better thermostability, while organic coating with the advantage of better compactness through polymerization and crosslinking usually provides

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better corrosion protection for metal substrates at relatively low temperature. Nanostructured poly (aniline-co-metaniolic acid) synthesized by copolymerization of aniline and metaniolic acid could be coated on carbon steel as organic corrosion resistance coating, and the corrosion resistance increased through decreasing the content of metaniolic acid. (Xing et al., 2014). Compared with normal water-based epoxy coating, the water-based epoxy coating added with 3-glycidoxypropyl trimethoxy silane exhibited better corrosion resistance performance (Bagherzadeh et al., 2012). Fluoropolymer film with outstanding chemical stability was also attempted on carbon steel via inductively coupled radio frequency plasma technique (Delimi et al., 2011). Polyurethane coating with the thickness of 50  $\mu\text{m}$ –100  $\mu\text{m}$  was also reported to present very effective protection against corrosion in 0.5 mol/L NaCl solution (Gonzalez-Garcia et al., 2007).

Except for pure inorganic corrosion resistance coatings and organic corrosion resistance coatings, inorganic-organic hybrid coatings are also gradually adopted by many researchers as corrosion resistance coating. The inorganic-organic hybrid coatings not only own the advantages of inorganic coating, but also the advantages of organic coating. For example, doped with inorganic polyphosphate, the hybrid polypyrrole coating coated on steel as corrosion resistance film exhibited good thermal stability with about 5% weight loss below 200 °C. The incorporation of polyphosphate obviously improved the coating thermotolerance (Mollahosseini and Noroozian, 2009). The electrodeposited polypyrrole films could also be modified with zinc. Zinc and its corrosion product blocked the pores of polymer, which increased the corrosion resistance performance of the films (Lehr and Saidman, 2013). The corrosion resistance of polypyrrole film could also be improved by adding magnetite and silane. The magnetite could stabilize polypyrrole film, and silane film could provide effective corrosion resistance performance (Montoya et al., 2014). And the polypyrrole coating could also be incorporated with TiO<sub>2</sub> to enhance corrosion protection (Lenz et al., 2003). Other hybrid coatings such as SiO<sub>2</sub>-polybenzoxazine nano-composite coating (Zhou et al., 2014), (Ce(NO<sub>3</sub>)<sub>3</sub>·6H<sub>2</sub>O)-doped hybrid silica coating (Santana et al., 2013), montmorillonite-doped polyaniline coating (Piomruen et al., 2014), metal-doped polyurushiol coating (Zheng et al., 2014), (polyaniline-ZnO)-doped epoxy coating (Mostafaei and Nasirpour, 2014) and silane-doped poly-methyl methacrylate coating (Sakai et al., 2012) were also reported to present better corrosion resistance performance than that of the coatings without doping. Therefore, inorganic-organic hybrid coatings usually offer better corrosion resistance for metal substrates at most time, while these hybrid coatings may also deteriorate the corrosion resistance occasionally due to the formation of more porous microstructure (Wang et al., 2011; Tian et al., 2010). The characteristics of these coatings are listed in Table 1.

Similar to 3.5 wt.% NaCl solution, Cl<sup>-</sup> is the critical factor intensifying the corrosion degree of carbon steel in geothermal water. While thermal stability and low toxicity for corrosion resistance coatings are also necessary besides corrosion resistance in geothermal environment. And recently, polysiloxane materials with the advantages of simple fabrication, thermal stability, low toxicity, water repellency and antifouling have been applied in many fields (Urata et al., 2013; Xiang et al., 2011; Brady, 2001; Wang et al., 2006). Therefore, a new kind of polysiloxane-ferroferic oxide corrosion resistance coating with high compactness, thermal stability, easy fabrication and low toxicity is required considering the low compactness of inorganic coatings, the weak thermal stability of organic coatings, the high pollution of most organic coatings fabrication and the complicated fabrication process of above-mentioned new-style coatings.

In this study, the corrosion resistance coating was fabricated via hydrothermal oxidation and dip coating of polysiloxane sol. Chemical compositions, microstructures, thermal stability, hydrophobicity, film equivalent thickness and corrosion resistance were investigated consecutively. And the corrosion process was described with potentiodynamic polarization curves, EIS and equivalent circuits. Compared with

carbon steel, the corrosion resistance performance of coated carbon steel was improved.

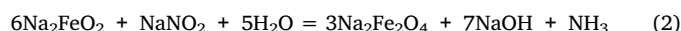
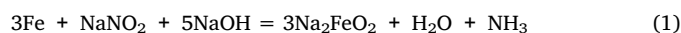
## 2. Experiments

### 2.1. Materials

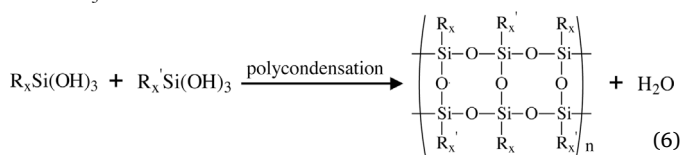
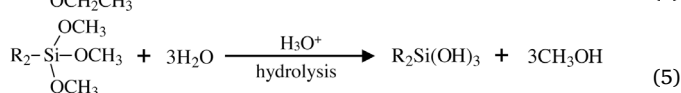
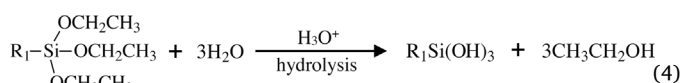
20# carbon steel with the size of 40 mm × 13 mm × 2 mm was used as substrate (Yangzhou Xiangwei Machinery Co. Ltd.) for test. Chemical reagents sodium hydroxide (NaOH), sodium nitrite (NaNO<sub>2</sub>) and ethyl alcohol (EtOH) bought from Jiangtian Chemical Technology Co. Ltd. are all analytically pure. Methyltriethoxysilane (MTES) and 3-Glycidoxypropyltrimethoxysilane (GPTMS) were purchased from Jinan Guobang Chemical Co. Ltd.

### 2.2. Sample preparation

All carbon steel substrates were polished and cleaned ultrasonically before being used. The solution used for hydrothermal oxidation was prepared by dissolving NaNO<sub>2</sub> and NaOH into distilled water. A reactor was used to hydrothermally oxidize carbon steel for 12 h at 130 °C. The chemical reaction equations can be written as follow:



The precursor solutions with different molar ratios of MTES:GPTMS: EtOH: H<sub>2</sub>O were stirred at least 2 h by magnetic stirring apparatus and aged for at least 24 h at room temperature before being used. The oxidized carbon steel substrates were dipped into precursor solutions with the speed of 5000  $\mu\text{m}/\text{s}$  and withdrawn from the solutions with the speed of 2000  $\mu\text{m}/\text{s}$ . This process was repeated three times before heat treatment at 120 °C for at least 3 h. After heat treatment the modified carbon steel was dipped again by the method above and kept in 120 °C for at least 3 h to an obtain thicker film. The composite coatings prepared with different molar ratios of MTES and GPTMS were named as Fe<sub>3</sub>O<sub>4</sub>-PGPTMS, Fe<sub>3</sub>O<sub>4</sub>-PMTES, Fe<sub>3</sub>O<sub>4</sub>-PMG-2:1, Fe<sub>3</sub>O<sub>4</sub>-PMG-1:1, and Fe<sub>3</sub>O<sub>4</sub>-PMG-1:2, respectively. The reaction process can be described by Eqs. (4)–(6).



R<sub>1</sub> refers to CH<sub>3</sub>, R<sub>2</sub> refers to CH<sub>2</sub>OCH<sub>2</sub>CH<sub>2</sub>O(CH<sub>2</sub>)<sub>3</sub>. R<sub>x</sub> and R<sub>x'</sub> refer to any one of R<sub>1</sub> and R<sub>2</sub>, respectively.

### 2.3. Characterization and measurement of the coatings

The chemical compositions of polysiloxane were characterized by Fourier transform infrared spectroscopy (FTIR) instrument (TENSOR 27, Bruker, Germany) and energy dispersive X-ray spectrum (EDX) (FEI, Nanosem 430, America).

The surface morphology was investigated by field emission scanning electron microscopy (FESEM) (FEI, Nanosem 430, America).

The thermal stability of prepared coatings was analyzed by TGA (METTLER TOLEDO, TGA/DSC1, Switzerland) with the heating rate of

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