



Characterization of corrosion scale formed on stainless steel delivery pipe for reclaimed water treatment



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ABSTRACT

To reveal corrosion behavior of stainless steel delivery pipe used in reclaimed water treatment, this research focused on the morphological, mineralogical and chemical characteristics of stainless steel corrosion scale and corroded passive film. Corrosion scale and coupon samples were taken from a type 304 pipe delivering reclaimed water to a clear well in service for more than 12 years. Stainless steel corrosion scales and four representative pipe coupons were investigated using mineralogy and material science research methods. The results showed corrosion scale was predominantly composed of goethite, lepidocrocite, hematite, magnetite, ferrous oxide, siderite, chrome green and chromite, the same as that of corroded pipe coupons. Hence, corrosion scale can be identified as podiform chromite deposit. The loss of chromium in passive film is a critical phenomenon when stainless steel passive film is damaged by localized corrosion. This may provide key insights toward improving a better comprehension of the formation of stainless steel corrosion scale and the process of localized corrosion. The localized corrosion behavior of stainless steel is directly connected with reclaimed water quality parameters such as residual chlorine, DO, Cl⁻ and SO₄²⁻. In particular, when a certain amount of residual chlorine in reclaimed water is present as an oxidant, ferric iron is the main chemical state of iron minerals.

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1. Introduction

Stainless steels are widely applied to processes for producing reclaimed water in water treatment plants throughout the world. They are used in diverse applications, such as delivery pipes, valves and water treatment equipment due to their corrosion resistance and durability (Ryan et al., 2002). Iron alloys contain a minimum of approximately 11% chromium which prevents the formation of rust in aqueous conditions (Sedriks, 1979). In general, AISI type 304 steel is the most commonly used austenitic stainless steel. It is commonly produced by standard cutting and turning processes for a cost-effective solution. These processes can cause strain-induced martensite to form at the machined surface of stainless steel, and improve their corrosion resistance (Martin et al., 2011). Although stainless steels have extremely good corrosion resistance, which is provided by a very thin surface Fe–Cr alloy oxide film, they can be

nevertheless susceptible to major modes of corrosion, including pitting corrosion, crevice corrosion, intergranular corrosion and stress corrosion cracking (Frankel, 1998; Hu et al., 2011; Laitinen, 2000; Lott and Alkire, 1989; Punckt et al., 2004; Zhang et al., 2015). When stainless steels are used in aggressive aqueous environments, pitting and crevice corrosion of stainless steels caused by chloride ions are common phenomena (Laitinen, 2000; Tian et al., 2014). Passive film is damaged and corrosion products form on the surface of iron alloy by electrochemical corrosion and oxidation reactions in the processes of pitting and crevice corrosion (Olsson and Landolt, 2001). Massive corrosion scales have occurred as a result of long-term accumulation of corrosion products. These lead to the failure of stainless steels, problems with equipment, water contamination and increased pumping costs (Sander et al., 1996). It is a crucial issue for water utilities. Inter-granular corrosion and stress corrosion cracking are serious problems for 304 stainless steels. These forms of corrosion are due to grain boundary sensitization when the material is exposed to nitric acid fluid or high temperature. The exposure results in the precipitation of chromium carbides at the grain boundary and the formation of chromium

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depletion zones (Hu et al., 2011; Martin et al., 2011; Ningshen et al., 2011; Yang et al., 2015). In water treatment plants, stainless steel corrosion behavior is usually caused by long-term exposure to water at normal temperature. When the inner surface of stainless steel is attacked by aggressive ions such as chloride, sulfate and thiosulfate in a neutral water environment, pitting and crevice corrosion of stainless steel are the two main forms of corrosion behavior that occur (Cai et al., 2010; Dornhege et al., 2007; Laitinen, 2000).

Most studies about corrosion products and scales have focused on iron and steel pipes used in water distribution systems (Gerke et al., 2008; Lin et al., 2001; McNeill and Edwards, 2001; Peng et al., 2010; Sander et al., 1996; Sarin et al., 2001; Tang et al., 2006; Yang et al., 2012). The physicochemical properties and formation processes of corrosion scales in new or old metallic pipes have been reported by numerous studies on water supply worldwide (Gerke et al., 2008; Sarin et al., 2001; Yang et al., 2012). Several researchers have investigated the structure and composition of corrosion scales in iron and steel pipes (Peng et al., 2010; Sander et al., 1996; Tang et al., 2006). Although they are sampled from different places in the pipes, these scales are usually composed of a corroded floor, a porous core, a shell-like layer and a top surface layer (Peng et al., 2010; Sarin et al., 2004b). The major chemical compositions of corrosion scales are oxides and hydroxides of iron, including magnetite (Fe_3O_4), hematite ($\alpha\text{-Fe}_2\text{O}_3$), maghemite ($\gamma\text{-Fe}_2\text{O}_3$), ferrous oxide (FeO), goethite ($\alpha\text{-FeOOH}$), lepidocrocite ($\gamma\text{-FeOOH}$), ferrous hydroxide ($\text{Fe}(\text{OH})_2$) and ferric hydroxide ($\text{Fe}(\text{OH})_3$). Minor substances like siderite (FeCO_3), green rust I and green rust II have been found in iron corrosion scales (Sarin et al., 2004b; Swietlik et al., 2012; Yang et al., 2012). Trace elements (Sr, As, Cr, V, U, Cd, Ni, and Mn) in corrosion scales have been detected in previous studies (Gerke et al., 2013, 2010; Peng and Korshin, 2011).

The formation of corrosion scales and process of corrosion in pipes is related to many factors. Pipe material, water quality and hydraulic conditions are the main causes. The effect of different water quality parameters including pH, residual chlorine, total organic carbon, conductivity, dissolved oxygen and alkalinity is coupled to mechanisms for inner metallic pipe material corrosion and corrosion scale formation (McNeill and Edwards, 2001; Sancy et al., 2010; Sarin et al., 2004a, 2004b). Uniform corrosion and localized corrosion are two traditional ways of classifying corrosion types. The former is a general way in which iron and steel pipes are attacked in water distribution systems (Sander et al., 1996). The latter includes pitting corrosion, crevice corrosion, intergranular corrosion and stress corrosion cracking, which are harmful to stainless steel (Dornhege et al., 2007; Punckt et al., 2004; Tian et al., 2014). It can impact on the service life of pipes. At present there is substantial research on corrosion of iron and steel pipes in water distribution systems. However, too few studies have focused on the corrosion of pipes used during the water treatment process, particularly stainless steel delivery pipes used in reclaimed water treatment (Sung et al., 2005). There are no studies in which the detailed morphological and physicochemical characteristics of stainless steel corrosion scales and corroded pipe have been described to identify the differences between stainless steel and iron.

In this paper, we focus on stainless steel corrosion scales and a corroded delivery pipe sampled from a reclaimed water plant. The fundamental objective of this work was to examine a stainless steel corroded pipe, and identify morphological and physicochemical characteristics of its corrosion scales using mineralogy and material science research methods. Meanwhile, the main corrosion behavior of a stainless steel pipe in use for 12 years was investigated, and water quality parameters relevant to the formation of corrosion

scales and process of pipe corrosion were analyzed in this work. The research results are expected to be useful for understanding the stainless steel pipe corrosion process in reclaimed water and will provide guidance for selecting stainless steel delivery pipes of water treatment.

2. Materials and methods

2.1. Samples collection of stainless steel corrosion scale and pipe coupon

Stainless steel corrosion scales and pipe coupons were sampled from a delivery pipe that linked to the clear well as shown in Fig. 1. The type 304 (composition: 0.08% C, 18.18% Cr, 1.20% Mn, 7.25% Ni, 0.016% S, 0.48% Si, with the remainder being Fe) pipe with diameter of 600 mm was in service for more than 12 years in Tianjin Water Recycling Plant. This plant was one of the earliest reclaimed water plants in China and was built as a demonstration project in 2002. The delivery pipe was removed after the waterworks ceased operations in that location. During operations, finished water with 5 mg/L chlorine concentration was diverted to the clear well through the stainless steel pipeline. The chlorine injection was directly welded onto the delivery pipe. The parameters of finished reclaimed water and the treatment processes of the reclaimed water plant are presented in Table 1. The corrosion scales were easily removed from the stainless steel pipe by tapping lightly with a rubber hammer. They were immediately stored in an airtight plastic bag for identification. The stainless steel corrosion scales were different sizes but all had a hemisphere pod shape. Fine scale powders were prepared by grinding samples after vacuum-freeze-dried pretreatment (Yang et al., 2012). Several stainless steel pipe coupons sampled from the delivery pipeline were cut into ingots with the dimensions of 10 mm × 10 mm × 2 mm. Corrosion products on the surface of these ingots were first removed with an iron brush. Then the ingots were cleaned in acetone for 15 min using an ultrasonic washer then naturally air dried at room temperature (Lu et al., 2014). Before the pretreatment procedure, photos were taken of samples using a Canon SX600 HS digital camera.

2.2. Inter-granular corrosion tests as per ASTM A 262 practice A

ASTM A262 practice A is used for the acceptance of austenitic stainless steels against inter-granular corrosion. In this test, the polished surface of the stainless steel specimen should be electrochemically etched in 10% oxalic acid solution for 1.5 min. The current density is 1 A/cm². Two samples were subjected to Practice A of ASTM A262. One was directly tested and another was tested after sensitizing heat treatments at 675 °C for 1 h. The electroetched microstructures of polished surface were identified with an optical microscope.

2.3. Scanning electron microscope-energy dispersive spectrometer (SEM-EDS)

The surface of corrosion scales and pipe coupons were observed using SEM (Zeiss MERLIN VP Compact, Germany). Their elemental composition was measured by coupled EDS.

2.4. X-ray diffraction (XRD)

The crystalline phase of corrosion scales and pipe coupons were examined using a RIGAKU (D-Max II) X-ray diffractometer with a Cu K α source setting at 200 mA and 40 kV. Corrosion scale powder was measured in step-scan mode with the 2 θ range of 10–90°. Step

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