

Corrosion behavior of tinplate in NaCl solution

XIA Da-hai¹, SONG Shi-zhe¹, WANG Ji-hui¹, BI Hui-chao¹, JIANG Yu-xuan², HAN Zhe-wen¹

1. School of Materials Science and Engineering, Tianjin University, Tianjin 300072, China;

2. School of Electronic Information Engineering, Tianjin University, Tianjin 300072, China

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Abstract: The corrosion process of tinplate in 0.5 mol/L NaCl solution was investigated using the electrochemical impedance spectroscopy (EIS), and the morphology and structure of the corrosion products were characterized by scanning electron microscope (SEM), scanning probe microscopy (SPM), X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS). The results showed that the resistance of tin coating, R_c , was essentially constant but the charge transfer resistance, R_{ct} , decreased by 2 orders of magnitude, which indicated that the tin coating was not seriously corroded while the carbon steel substrate was corroded continuously. The corrosion of tinplate in 0.5 mol/L NaCl solution was mainly the dissolution of carbon steel substrate because of the defects in the tin layer and the corrosion product was mainly γ -FeOOH.

Key words: tinplate; corrosion behavior; electrochemical impedance spectroscopy; NaCl solution

1 Introduction

Tinplates, one of the most common packaging materials, are used in more than 80% of cases though the new alternative materials such as aluminum and chromated steel sheet are increasingly used by the canning industry [1–4]. Tinplate is a light gauge, cold reduced, low-carbon steel sheet or strip, coated on both sides with commercially pure tin, combining the strength and formability of steel and the corrosion resistance with good appearance of tin. Tinplate has become one of the dominant materials for food cans.

However, there are significant problems related to the use of tinplate cans in corrosive food products, such as corrosion failure, loss of seal integrity, or discoloration problems that cause their rejection by the consumer [5–9]. In addition, other studies [10,11] also indicate that high level of tin in food product may lead to food safety problem. Though tin is not considered to be a poisonous metal, very large doses can produce serious digestive disturbances [11,12]. The corrosion process of pure tin is relatively simple due to its homogeneity. Numerous studies have been focused on the corrosion behaviors of pure tin [13–20]. Anodic behaviors of tin showed active-passive transition in the presence of citrate buffer solution [15,20], Na_2CO_3 solution in the

absence or presence of Cl^- or I^- ions [17], sodium borate solutions containing halide ions and some inorganic inhibitors [18], NaOH solution [13], citric acid electrolytes [19]. But the corrosion process of tinplate is complicated due to its stratified structure and heterogeneity. In an acid medium, it is advantageous that tin is the sacrificial member of the couple, providing protection to the steel substrate while corroding at a certain rate itself [1,2]. Very few studies on the corrosion behaviors of tinplate have been reported, and the corrosion behaviors in neutral media are still uncertain. NaCl commonly exists in seafood and cured food products. Therefore, a study on the corrosion behavior of tinplate in NaCl solution has noted significance [3,21,22]. The different industries involved in tinplate cans (tinplate producers, can manufactures, food canners, etc.) and health authorities are in need of information about the behavior of this material.

Tinplate is a coating metal system and its electrochemical signal during corrosion process can be detected by the electrochemical impedance spectroscopy (EIS) technique, hence the anticorrosion performance of tinplate can be quantitatively and semi quantitatively evaluated. Though in most cases, organic-coated tinplates are used as food cans materials, uncoated tinplate is used as the research object in order to study the corrosion form and corrosion process beneath the

organic coating. The objective of the present work is to understand the corrosion mechanism of tinplate in NaCl solution by the methods of EIS, X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS).

2 Experimental

2.1 Materials

All the tinplate sheets used in this work were provided by the ORG Canmaking Company. The tinplate was a low carbon steel with tin coated on both sides by electrodeposition and the thickness of the tin coating on both sides was about 3 μm . The samples (70 mm $\times$ 70 mm) were degreased by ethanol and dried before being exposed to NaCl solution.

2.2 Electrochemical measurements

The potentiodynamic polarization and EIS measurements were carried out in an electrolytic cell using a VersaSTAT 4 electrochemical workstation (Princeton Applied research, USA) and VersaStudio control software. The potentiodynamic polarization experiments were performed at a scanning rate of 0.1667 mV/s. EIS measurements were performed at the open-circuit potential with a 5 mV amplitude signal and the applied frequency ranged from 100 kHz to 0.01 Hz. A three-electrode cell with the tinplate as the working electrode (WE), an antimony electrode as the reference electrode (RE) and a commercial ruthenium–titanium electrode as the counter electrode (CE) was used. The sample was exposed to 0.5 mol/L NaCl solution at room temperature of (25 $\pm$ 2) $^{\circ}\text{C}$ and examined periodically by EIS technique. The data acquired were fitted by ZSimpWin software.

2.3 X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS)

The compositional line profiles of the cross section were characterized by an energy dispersive X-ray analyzer (EDAX Genesis). The crystal structure was identified using an X-ray diffractometer (RIGAKU/DMAX2500, Japan).

The XPS spectra were taken using a commercial X-ray photoelectron spectroscope (PHI1600ESCA XPS). A survey spectrum was first recorded to identify all elements present on the surface, and then high resolution spectra of the following regions were recorded: Fe 2p, O 1s, and Sn 2d. A Shirley background subtraction was made to obtain the XPS signal intensity.

2.4 Scanning electron microscopy (SEM) and scanning probe microscopy (SPM)

SEM analyses were carried out on a field-emission scanning electron microscope (FE-SEM S4800; Hitachi,

Japan). For cross-section characterization, all of the samples were prepared by the Buehler Company Limited (USA). The morphology and microstructure of the tinplate were characterized on a scanning probe microscopes (AJ-IIIa, China).

3 Results

3.1 Potentiodynamic polarization

Figure 1 shows the effect of concentration of NaCl solution on polarization behaviour of tinplate at room temperature. No obvious active–passive transition potential peak was observed in NaCl solutions, indicating highly aggressive behavior in NaCl solutions. The anodic current was significantly increased by increasing the NaCl addition.

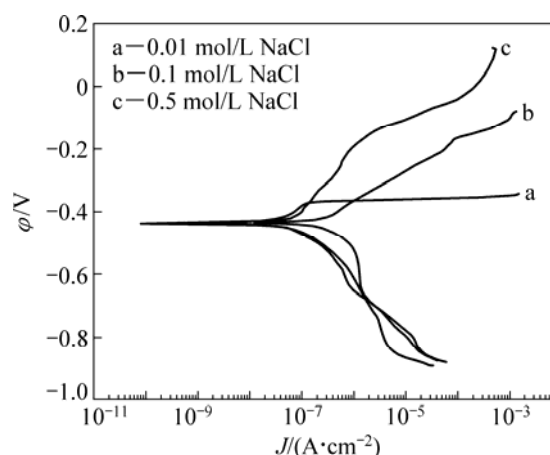


Fig. 1 Potentiodynamic polarization curves of tinplate in 0.01 mol/L NaCl solution (a), 0.1 mol/L NaCl solution (b), and 0.5 mol/L NaCl solution (c)

3.2 EIS analysis

Figure 2 shows the typical EIS plots of tinplate exposed to 0.5 mol/L NaCl solution over a period of 16 d. Figure 3 gives the electrochemical equivalent circuit and the fitting results. This parallel circuit was selected because the corrosion occurring in the interface of tin/carbon steel was considered uniform, which was confirmed by SEM observations.

After 1 h exposure, the Bode plot showed a characteristic with two time constants, indicating that the electrolyte had permeated through the defects of the tin coating and double layer capacitance was formed on the substrate metal interface. The time constant at higher frequency was a result of tin coating interface capacitance (Q_c) and surface pore resistance of tin coating (R_c); the time constant at low frequency was a result of double layer capacitance (Q_{dl}) and charge transfer resistance (R_{ct}). R_{ct} reflected the difficulty level of charge transfer process and corrosion rate [23]. As exposure time was prolonged, the radius of the

capacitance arc decreased. The continued decrease of the impedance indicated a promotion of corrosion in the tinplate. The low frequency impedance $|Z|_{0.01\text{Hz}}$ was approximately equal to the charge transfer resistance and was found to correlate with visually observable coating/metal system degradation. It was found that $|Z|_{0.01\text{Hz}}$ value decreased from 137.1 to 14.96 $\text{k}\Omega\cdot\text{cm}^2$, indicating that the protective performance of tinplate was also lessened.

The R_c and R_{ct} values obtained from the electrochemical equivalent circuit are shown in Fig. 3. The R_c value varied from 296.6 to 251.8 $\Omega\cdot\text{cm}^2$ during the whole exposure time, and it was on the same order of magnitude. But the R_{ct} value decreased by 2 orders of magnitude, from 178.4 to 15.4 $\text{k}\Omega\cdot\text{cm}^2$, indicating that the carbon steel substrate was corroded continuously. Because R_{ct} was inversely proportional to corrosion rate, the decrease of R_{ct} indicated that the corrosion on carbon steel substrate became more and more serious.

3.3 Surface and cross section morphology

Figure 4 shows SEM image and compositional line profiles of tinplate before and after being exposed to 0.5 mol/L NaCl solution for 16 d. Before exposure, a clean surface with no corrosion products was observed on the surface of tinplate (Fig. 4(a)). But after being exposed to 0.5 mol/L NaCl solution for 16 d, many acicular corrosion products were observed on the surface (Fig. 4(b)). Moreover, we can also see the change in the cross section image and compositional line profiles. The

compositional line profiles were probed by energy-dispersive X-ray spectroscopy (EDS), mainly exhibiting Fe, Sn and O signals across the cross section. Tinplate is made of a low-carbon steel sheet, coated on both sides with pure tin, so a 3 μm -tin coating was observed. The compositional line profiles on the cross section of tinplate before being exposed to NaCl solution (Fig. 4(c)) showed that Fe content was high but Sn content was low in the region of substrate; Sn content was high but Fe content was low in the region of tin coating. Figure 4(d) shows the cross-section morphology and compositional line profiles of tinplate after being exposed to NaCl solution for 16 d. The compositional line profiles results showed that Sn content was still high in the region of tin coating, and the structure of tin coating had no evident change. But it was remarkable that the interface of the tin/carbon steel was seriously corroded, but tin coating showed no obvious signs of failure.

3.4 XRD and XPS analyses

After immersion in NaCl solution for 16 d and then exposure to air for 1 d, the corrosion product was analyzed using XRD. XRD pattern of the corrosion products is shown in Fig. 5. The diffraction peaks of the samples can be indexed to the phases of NaCl and lepidocrocite ($\gamma\text{-FeOOH}$). NaCl was detected because there was residual NaCl on the surface of the tinplate. $\gamma\text{-FeOOH}$ was the main corrosion product. The results are similar to those reported about the corrosion products of carbon steel in aquiferous environment containing Cl^-

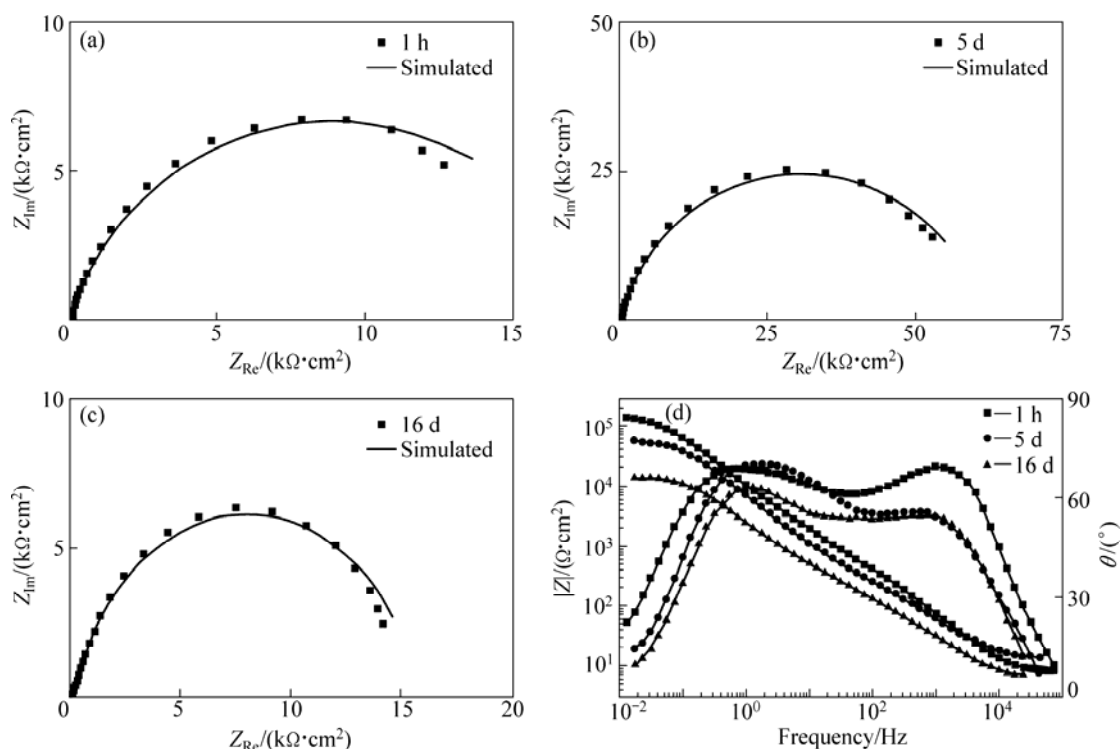


Fig. 2 EIS plots of tinplate after exposure to 0.5 mol/L NaCl solution for 1 h (a), 5 d (b) and 16 d (c) (solid line are fitting results) and Bode plot (d)

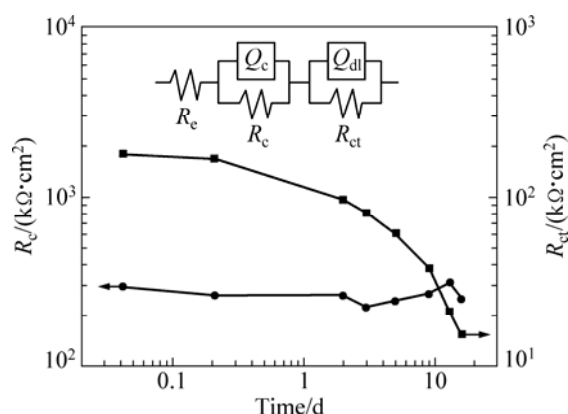


Fig. 3 Evolution of R_e and R_{ct} values of electrochemical equivalent circuit as function of exposure time and established electrochemical equivalent circuit (R_e : electrolyte resistance; Q_c : capacitance of tin coating; R_c : resistance of tin coating; Q_{dl} : substrate metal double layer capacitance; R_{ct} : charge transfer resistance).

[24,25]. According to the results of HUANG et al [13], the corrosion films on pure tin exposed to NaCl solution were composed of $\text{Sn}(\text{OH})_2$, SnO_2 and/or SnO . Those three corrosion products were not detected on the tinplate, indicating that the quantity of them was possibly so small. This also indicated that the corrosion of tinplate was different from pure tin.

X-ray photoelectron spectroscopy (XPS) was used to further analyze the chemical composition of the corrosion film. The XPS spectra of the unexposed and

corroded tinplate are shown in Fig. 6. Both the spectra showed the presence of C, Sn, O, but the appearances of Na, Cl and Fe were only observed in the exposed tinplate, which indicated that the corrosion products appeared and covered on the tinplate due to exposure to NaCl solution. The appearance of Cr was observed in the unexposed tinplate, which was associated with chromating passivation of the tinplate. The atomic concentrations of C, O, Sn and Cr before immersion measured by XPS were 51.2%, 34.8%, 9.4%, 4.6%, respectively (see Table 1). The presence of carbon may be due to the accumulation of contaminants. After immersion in NaCl solution for 16 d and exposure to air for 1 d, Sn content decreased to 0.4%, which can be due to the coverage of the corrosion products; Fe content increased to 14.2%, indicating that the corrosion products primarily was an oxide of iron.

Figure 7 shows the high-resolution XPS spectra of the O 1s, Fe 2p and Sn 2d regions for the tinplate. Figures 7(a) and (b) show the spectra of O 1s region before immersion and after immersion. Both the O 1s regions were composed of two peaks. The dominant peaks at 529.5 eV and 531 eV were attributed to O^{2-} [7,15]. The first one at 529.5 eV could be attributed to SnO or SnO_2 [15], and the second one at 531 eV can be attributed to hydroxide of $\text{Sn}(\text{OH})_2$ [15] or FeOOH . It should be noticed that a SnO or SnO_2 oxide film will form on the surface when tin is exposed to the air [14]; and $\text{Sn}(\text{OH})_2$ possibly exists on the surface because of the exposure of tin to NaCl solution.

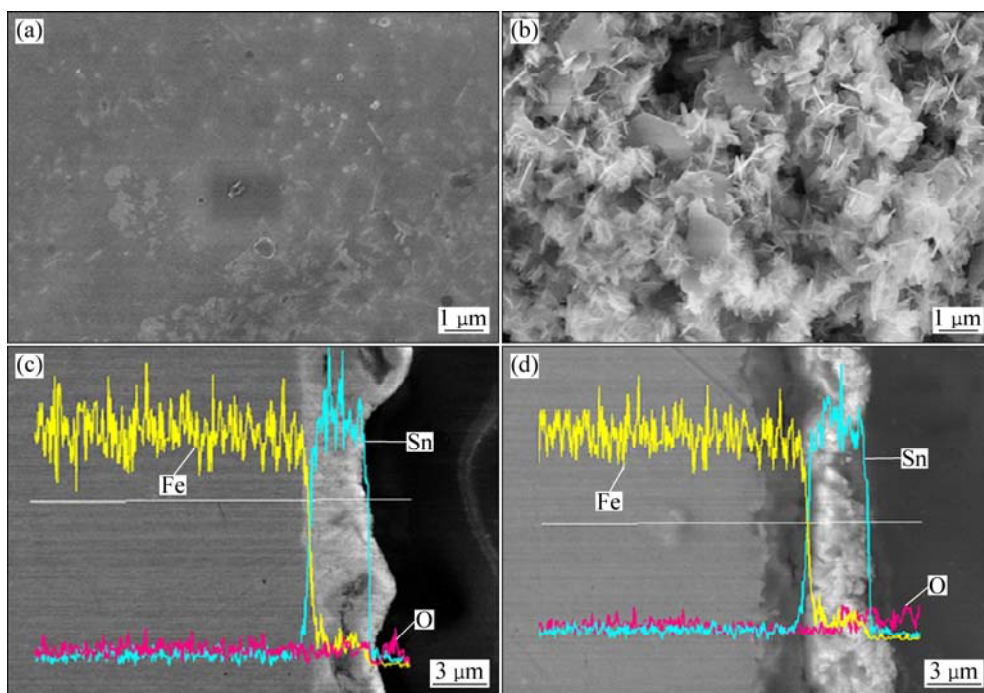


Fig. 4 SEM surface images and corresponding compositional line profiles of the cross section of tinplate before (a, c) and after exposure to 0.5 mol/L NaCl solution for 16 d (b, d) probed by EDS spectroscopy scanning along white line

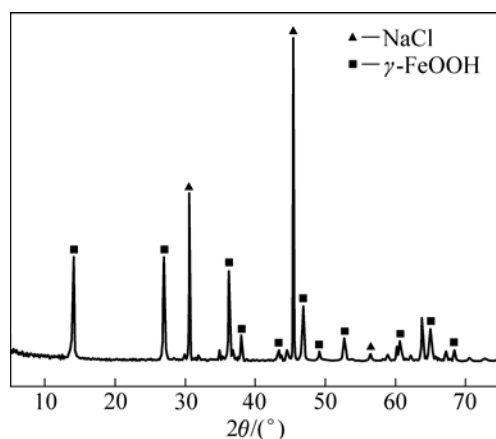


Fig. 5 XRD pattern of corrosion product on surface of tinplate exposed to 0.5 mol/L NaCl solution for 16 d and then exposed to air for 1 d

Figures 7(c) and (d) show the spectra of Fe 2p regions. The dominant peak at 715.5 eV was attributed to Fe(II), possibly associated with the oxide of iron, associated with a small amount of exposed iron in the tin coating. The dominant peaks at 711.5 eV and 724.3 eV were attributed to Fe(III) [7,26], which mainly exist as FeOOH [25,27].

The deconvoluted Sn 3d spectra show three components (Figs. 7(e), (f)) corresponding to tin in the forms of Sn⁰, Sn(II) or Sn(IV), and the low-intensity line at 484.3 eV could be attributed to metallic tin (Sn⁰) in accordance with BE values (484.4±0.5) eV presented by other authors[6,7,15,17]. Unfortunately, the obtained XPS spectra did not allow to distinguish clearly the Sn(II) oxidation state from Sn(IV) [15]. In summary, besides FeOOH, a very small amount of SnO or SnO₂ or a combination of these two existed on the surface before exposure; comparing to a very small amount of Sn(OH)₂, SnO or SnO₂ possibly existed on the surface when tin exposed to NaCl solution.

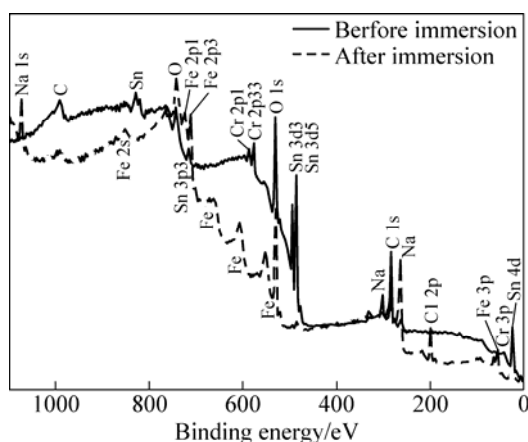


Fig. 6 XPS spectra acquired from surface of tinplate before and after exposure to 0.5 mol/L NaCl solution for 16 d

Table 1 Composition of tinplate before and after immersion in 0.5 mol/L NaCl for 16 d (measured by XPS)

Sample	Composition/%						
	C	O	Sn	Cr	Fe	Na	Cl
Before immersion	51.2	34.8	9.4	4.6	—	—	—
After immersion	30.7	36.5	0.4	—	14.2	9.3	8.9

4 Discussion

In order to investigate the corrosion mechanism, we studied the nanometer-level surface morphology of tinplate before exposure to NaCl solution, which is shown in Fig. 8. In scanning probe microscopy (SPM) small scale images (~3 μm²), we can see an uneven surface structure (Fig. 8(a)), indicating that the tin coating was defective. The particle size was about 22 nm in diameter (Fig. 8(b)). The defect in the coating is one of the reasons leading to corrosion of carbon steel.

Based on the SPM observations, and EIS, XRD and XPS analyses, the corrosion process of tinplate in 0.5 mol/L NaCl solution is divided into two steps, which are illustrated schematically in Fig. 9.

O₂ gas from the air come into the solution surface of the carbon steel. In the defects, the Sn-Fe alloyed layer (FeSn₂) was exposed to aggressive solution but the layer was so thin and carbon steel was soon exposed to the electrolyte. Due to the potential difference between tin/carbon steel substrate and the defects in the tin coating, corrosion occurs at the defects on the surface of the tinplate because of highly exposed carbon steel substrate. The corrosion occurs when the cathodic reaction is oxygen reduction on the tin coating [28]. Electrons flow from the carbon steel, which serves as the anode (Reactions (a) and (2)), to the oxygen-rich region on the surface, which serves as the cathode (Reaction (3)).

Anodic reaction:



Cathodic reaction:



Under the condition of high amount of Cl⁻, the existence of Cl⁻ is conducive to the formation of FeOOH [29]. The possible forming process of γ-FeOOH is possibly as follows [30–33]:



Under dry condition,



The SEM morphology showed that some acicular

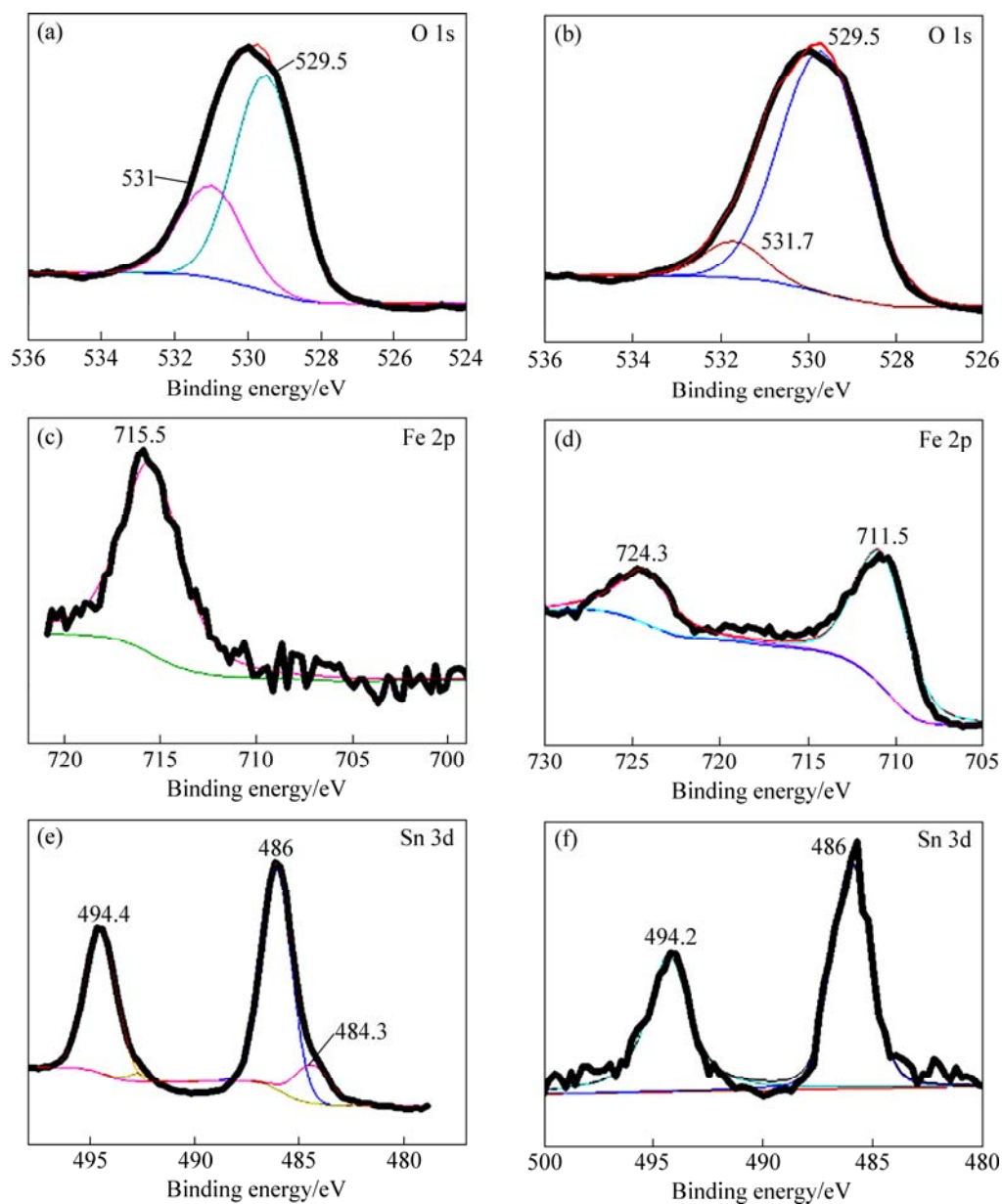


Fig. 7 XPS spectra of tinplate surface before (a, b, c) and after exposure (d, e, f) to 0.5 mol/L NaCl solution for 16 d: (a, b) O 1s; (c, d) Fe 2p; (e, f) Sn 3d

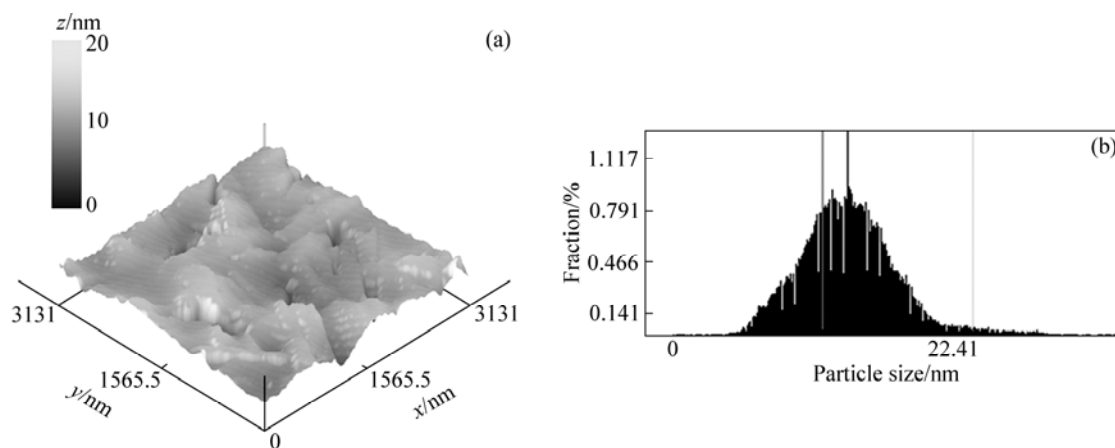


Fig. 8 SPM images of tinplate surface before exposure: (a) 3-dimensional morphology; (b) Particle size distribution

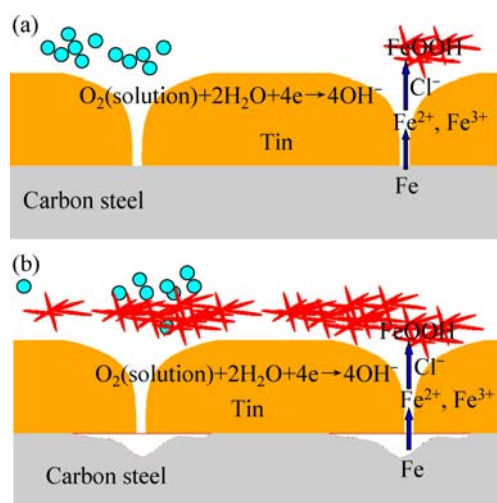


Fig. 9 Schematic representations of corrosion process for tinplate exposed to 0.5 mol/L NaCl solution: (a) Initial stage; (b) After serious corrosion

γ -FeOOH crystals accumulated on the electrode surface after being exposed to NaCl solution for 5 d and there were more crystals after 16 d. Moreover, the cross-section morphology showed the carbon steel substrate was corroded after 16 d. As mentioned previously, the formation of γ -FeOOH was proved by XRD and XPS.

5 Conclusions

1) The SEM images showed that the tin coating was not seriously corroded while the carbon steel substrate was corroded continuously.

2) The corrosion product formed on tinplate after exposure to 0.5 mol/L NaCl solution for 16 d was γ -FeOOH. This was further confirmed by the XPS technique.

3) Due to the potential difference between tin and carbon steel in NaCl solution, and also the defects in the tin coating, corrosion occurs at the defects on the surface of the tinplate because of high exposed carbon steel substrate. The corrosion of tinplate in 0.5 mol/L NaCl solution is the dissolution of carbon steel substrate.

Acknowledgements

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镀锡薄钢板在 NaCl 溶液中的腐蚀行为

夏大海¹, 宋诗哲¹, 王吉会¹, 毕慧超¹, 蒋雨轩², 韩哲文¹

1. 天津大学 材料科学与工程学院, 天津 300072;

2. 天津大学 电子信息工程学院, 天津 300072

摘 要: 应用电化学阻抗谱技术研究镀锡薄钢板在 0.5 mol/L NaCl 溶液中的腐蚀过程, 结合 SEM、SPM、XRD、XPS 技术表征腐蚀产物的形貌和腐蚀产物的结构。结果表明: 腐蚀过程中镀锡层的电阻 R_c 值基本不变而电荷转移电阻 R_{ct} 值下降了 2 个数量级, 表明镀锡层基本没有发生腐蚀而基底金属碳钢则不断遭到腐蚀。镀锡薄钢板在 0.5 mol/L NaCl 溶液中的腐蚀类型主要是对镀锡层缺陷处暴露的基底金属碳钢的腐蚀, 其腐蚀产物主要是 $\gamma\text{-FeOOH}$ 。

关键词: 镀锡薄钢板; 腐蚀行为; 电化学阻抗谱; NaCl 溶液

(Edited by YANG Hua)