



X-ray photoelectron spectroscopy investigation of commercial passivated tinplate surface layer



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ABSTRACT

X-ray photoelectron spectroscopy (XPS) combined with the low energy Ar⁺ sputtering technique has been used to investigate the chemical compositions and chemical states of elements at different depths of commercial passivated tinplate surface layer. It is found that Cr₂O₃, SnO, Cr(OH)₃, metallic Sn and a small amount of metallic Cr have been mixed in this layer. According to peak fitting and relative sensitivity factor method, the concentrations of elements in various chemical environments on different depth planes of the passivated tinplate surface layer have been obtained.

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1. Introduction

Tinplate is sheet steel covered with a thin layer of tin. By far the largest application of tinplate is in packaging. It is ideally suited for this purpose, by virtue of it being non-toxic, light in weight, strong, and corrosion resistant. It is easily formed, soldered and welded. In addition, it also provides an excellent printing surface, and can be fully degradable. So tinplate is widely used for packaging food-stuffs, beverages, oils, grease, paints, powdered, polishes, waxes, chemicals and many other products [1,2].

Chromium-based treatments are currently used for the passivation of tinplate, which produces a protective surface layer [3,4]. This passivation film is effective against the formation of tin oxides and corrosion by food media, offering a good substrate for the adhesion of lacquer.

Due to the importance of tinplate surface passivation, researches in this area have been carried out by different teams [5,6]. But the detailed surface layer structure has seldom been analyzed carefully. As a fundamental work, the knowledge of surface layer structure ensures a deep understanding of passivated tinplate and a valuable guidance to improve productive technology.

X-ray photoelectron spectroscopy (XPS) combined with the low energy Ar⁺ sputtering technique is one of the most popular surface spectroscopic techniques to investigate the depth profiles of the material surface [7,8]. It can be employed to analyze

chemical composition at different depths of material surface, layer and supply the information about chemical environments of elements. XPS behaves better in non-crystalline material analysis than X-ray or electron diffraction. And it also has advantage over energy disperse spectroscopy (EDS) in the area of super thin film analysis. This method is suitable for thin films with the thickness of tens of nanometers, voiding difficult transmission electron microscopy (TEM) sample preparation when TEM analysis used in the area with such a scale. So XPS is an ideal method to research the surface layer structure of passivated tinplate.

The aim of this paper is to investigate the surface layer structure of passivated tinplate by XPS depth profiling. Through careful analysis of experimental data, the depth distribution of various elements in different chemical environments has been obtained. On the basis of this, the surface layer structure of passivated tinplate is revealed.

2. Experimental

Commercial tinplates made by Baosteel with coating mass on both sides of 2.8 g/m² were investigated. The tinplates were passivated by means of a dip treatment in sodium dichromate solution (25 g/L) at 44 °C.

XPS measurements were conducted using Quantera SXM^{XP} system made by ULVAC-PHI cooperation with monochromatic Al K α (1486.6 eV) radiation. Survey scans and high resolution scans were performed at the pass energy of 112 eV and 26 eV, respectively. The instrumental binding energy scales have been calibrated using standard samples of pure copper (Cu), silver (Ag) and gold (Au)

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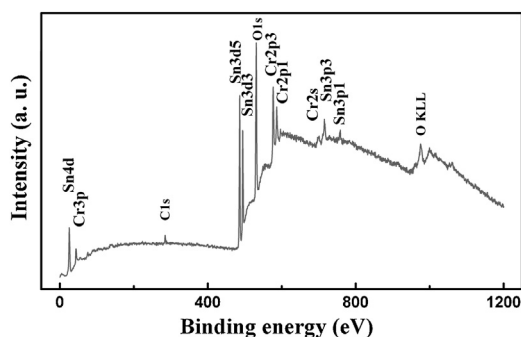


Fig. 1. XPS survey spectrum acquired on the surface of the passivated tinplate sample.

according to ISO 15472. The base vacuum of the analysis chamber is better than 2×10^{-9} Torr. XPS depth profiling was carried out by argon ion bombardment (AIB). An ion accelerating voltage of 2 kV was used to analyze the surface layer structure of coating, and the sputtering rate of pure Sn is about 21 nm/min.

3. Results and discussion

Fig. 1 displays the XPS survey spectrum acquired on the surface of the passivated tinplate sample after cleaning. From this figure, it can be seen that there are only oxygen, tin, chromium and carbon on the surface of the sample. The weak carbon signal in this figure comes from adventitious hydrocarbon on the surface.

XPS depth profile has been used to investigate the inner region of the surface layer. It is confirmed that carbon only exists on the topmost surface of the sample, since carbon signal disappears after a very light (about 0.1 min) Ar^+ sputtering. From Sn 3d XPS spectra acquired at different sputtering time, it is obvious that the valence state of Sn on the surface is different with which in the coating layer, as shown in Fig. 2. In this figure, it can be noticed that there is a shoulder peak beside the main peak in the curve corresponding to the sputtering time of either 0 min or 0.4 min. By peak fitting, as shown in Fig. 3, Sn $3d_{5/2}$ peak corresponding to either 0 min or 0.4 min has two components. The component with the binding energy of 484.7 eV is attributed to Sn- β , while the one with the binding energy of 486.2 eV can be attributed to Sn^{2+} in SnO [9]. From Fig. 3, it is clear that Sn^{2+} component is the major part of Sn on the surface, while Sn- β exists on the surface with a small amount. After Ar^+ sputtering for 0.4 min, Sn- β becomes the major part of Sn, while Sn^{2+} concentration reduces sharply. Finally, Sn^{2+} signal vanishes after Ar^+ sputtering for 0.8 min. With the increasing

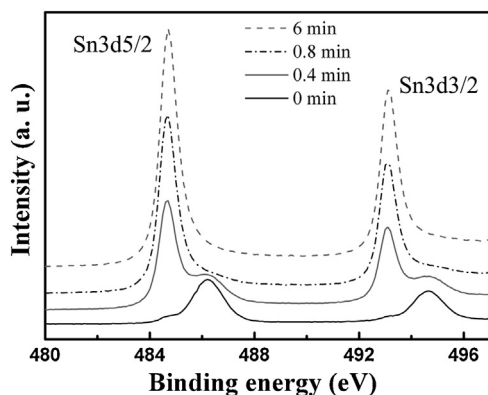


Fig. 2. Sn 3d XPS spectra of the passivated tinplate acquired at different Ar^+ sputtering time.

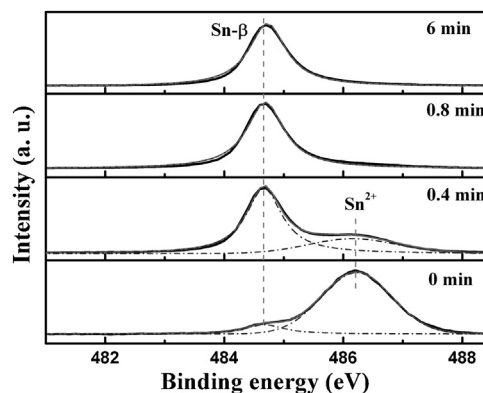


Fig. 3. Analysis of Sn $3d_{5/2}$ peaks acquired at different Ar^+ sputtering time.

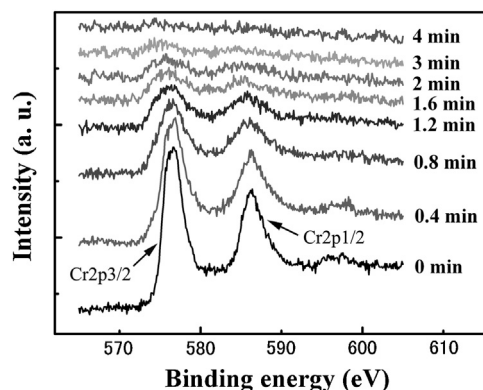


Fig. 4. Cr 2p XPS spectra of the passivated tinplate acquired at different Ar^+ sputtering time.

of sputtering time after that, Sn in the whole coating layer reveals metallic Sn.

Fig. 4 reveals the variation of Cr 2p peak with sputtering time. Cr signal becomes more and more weak with the continuance of Ar^+ sputtering, and cannot be detected at the sputtering time of 4 min. Detailed analysis of Cr $2p_{3/2}$ XPS depth profile is shown in Fig. 5. Because the signal-to-noise ratios of the curves after sputtering time of 1.6 min are so low that it is difficult to carry out peak fitting precisely, the Cr $2p_{3/2}$ curves at the sputtering time of 0 min, 0.4 min, 0.8 min and 1.6 min are chosen to analyze in Fig. 5. The curve acquired at the sputtering time of either 0.4 min or 0.8 min can be fitted to one symmetric peak with the binding energy of 576.2 eV. Considering there are only one symmetric Cr $2p_{3/2}$ peak and one symmetric O 1s peak (shown later in this article) in XPS

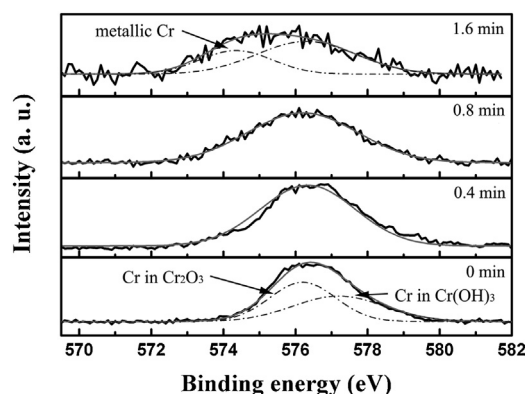


Fig. 5. Analysis of Cr $2p_{3/2}$ peaks acquired at different Ar^+ sputtering time.

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