

Electrosynthesis and physicochemical properties of α -PbO₂-CeO₂-TiO₂ composite electrodes

Bu-ming CHEN¹, Zhong-cheng GUO^{1,2}, Rui-dong XU¹

1. Faculty of Metallurgical and Energy Engineering,
Kunming University of Science and Technology, Kunming 650093, China;
2. Kunming Hengdera Science and Technology, Kunming 650105, China

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Abstract: In order to investigate the effect of solid particles dopants on physicochemical properties of α -PbO₂ electrodes, α -PbO₂ composite electrodes doped with nano-TiO₂ and nano-CeO₂ particles were respectively prepared on Al/conductive coating electrodes in 4 mol/L NaOH solution with addition of PbO until saturation by anodic codeposition. The electrodeposition mechanism, morphology, composition and structure of the composite electrodes were characterized by cyclic voltammogram (CV), SEM, EDAX and XRD. Results show that the doping solid particles can not change reaction mechanism of α -PbO₂ electrode in alkaline or acid plating bath, but can improve deposition rate and reduce oxygen evolution potential. The doping solid particles can inhibit the growth of α -PbO₂ unit cell and improve specific surface area. The diffraction peak intensity of α -PbO₂-CeO₂-TiO₂ composite electrode is lower than that of pure α -PbO₂ electrode. The electrocatalytic activity of α -PbO₂-2.12%CeO₂-3.71%TiO₂ composite electrode is the best. The Guglielmi model for CeO₂ and TiO₂ codeposition with α -PbO₂ is also proposed.

Key words: Al; lead dioxide; composite electrodes; electrocatalysis; physicochemical property

1 Introduction

Composite coating is a special coating formed in the process of metal or metal oxide layer with inclusion of one or several insoluble solid particles by metal or metal oxide electrodeposition method [1]. YEO et al suggested that mixed oxide doped with some ions could evidently reduce the oxygen evolution overpotential and pointed out that the overpotential oxygen-transfer reactions and oxygen evolution were correlated because the two processes were both discharged of water anode [2,3]. Since 1996, some composite electrodes have been prepared by composite electrodeposition of PbO₂, Ti₂O₃ as matrix metal oxides and RuO₂, Co₃O₄ catalytic particles as disperse phases [4], and applied to the oxygen evolution of electro-catalysis materials. The incorporation of solid particles into PbO₂ led to significant change of the electrocrystallization morphology. Furthermore, the contents of dispersed

phases in the composite electrodes depend on electrolyte compositions and electrodeposition conditions [5].

Nano-composite coating is mixed with nano-particles in the size range of 0.1–100 nm, which have higher hardness, wear resistance, friction reduction and corrosion resistance owing to the small size effect, surface effect, quantum size effect and quantum tunnel effect of nano-particles. Nano-TiO₂ particle as a photocatalyst is widely used in air purification, cleaning, and sterilization because of its good chemical stability, insolubility, non-toxicity and low cost. Rare earth has a wide range of catalysis and co-catalysis because of its unique 4f electronic structure, physical and chemical properties. The course of electrocrystallization will be changed when cerium dioxide particles are embedded in the coating, which promotes the crystal plane preferring to orientation growth. The microstructures for coatings are more uniform and compact, so the corrosion resistance is improved [6].

In order to get a kind of composite material with

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Corresponding author: Zhong-cheng GUO; Tel: +86-871-8352598; E-mail: guozhch@vip.163.com

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high electrochemical activity, Al/conductive coating/ α -PbO₂-CeO₂-TiO₂ composite electrodes were prepared by anodic codeposition in an alkaline bath. The effects of solid particles dopants on physicochemical properties of α -PbO₂ electrodes were investigated. The electro-deposition mechanism, morphology, composition and structure of the composite electrodes were characterized.

2 Experimental

2.1 Preparation of composite electrodes

Aluminum (Al) in the dimensions of 50 mm×25 mm×2 mm was used as the substrate, which was first roughened by sand-blasting, degreased and chemically etched, and then coated by a conductive coating. The procedure was described as follows: 1) the conductive solution was applied to the substrate by brushing; 2) the substrate was surface dried under ultraviolet lamp; 3) the substrate was dried in an electricity box at 423 K for 2 h. The undercoating produced in this research was 20–30 μ m thick, and the details were presented in Ref. [7]. Thereafter, the composition and process conditions of the PbO₂ plating bath were as follows: 4 mol/L NaOH solution saturated with litharge PbO(s) (the soluble PbO species were HPbO₂[−]), pH>14, anode current density of 0.3–1.0 A/dm², mildly stirring using a magnetic stirrer, bath temperature of 20–50 °C, 0–20 g/L TiO₂ grain (rutile, the average particle size of 30 nm), 0–20 g/L CeO₂ grain (the average particle size of 50 nm), and electroplating time of 4 h. The bath was dispersed for 30 min by the ultrasonic device before electrodeposition in order to assure that TiO₂ and CeO₂ particles were dispersed in oxide substrate. Finally, Al/conductive coating/ α -PbO₂-CeO₂-TiO₂ composite electrode was obtained.

2.2 Characterization of composite electrodes

The electrochemical performance of Al/conductive coating/ α -PbO₂-CeO₂-TiO₂ composite electrode was measured with the three-electrode cell. The composite electrode was used as the working electrode, Hg/Hg₂Cl₂ (KCl, saturated) was used as the reference electrode, and graphite was used as the auxiliary electrode. Cyclic voltammetric curves were measured in an alkaline bath. The alkaline bath, called S1, contained 4 mol/L NaOH solution saturated with litharge PbO(s) at 40 °C in the potential range of 0–1.4 V. The acid bath, called S2, contained 30% Pb(NO₃)₂ (pH=1.5). The bath temperature was kept at 60 °C in the potential range of 0–2.1 V. The surface morphology of the composite electrodes was characterized by SEM (XL30 ESEM, Philip, Holland). The composition of the composite electrode was obtained by EDS (PHOENIX, EDAI, USA). The phase structure of the composite electrode

was studied by X-ray diffraction (using Co K _{α} radiation, X'pert Highscore produced by Netherlands Philips Instrument Company).

3 Results and discussion

3.1 Cyclic voltammetry curves

3.1.1 Cyclic voltammetry curves of composite electrode in alkaline solution

The cyclic voltammetry curves in 4 mol/L NaOH solution and in S1 solution at 40 °C are shown in Fig. 1. Figure 1(a) shows that an oxidation peak appears at 0.2 V vs SCE, which may be a result of the redox transition PbO/PbO₂ (PbO+2OH[−]−2e=PbO₂+H₂O), and an oxygen evolution when the potential is over 0.7 V vs SCE. As shown in Fig. 1(b), the oxidation peak intensity does not change when the oxygen evolution potential changes. It can be seen from Fig. 1(c) and Fig. 1(d) that the original potential of α -PbO₂ is 0.20 V vs SCE [8]. Two anodic current density peaks at ϕ_{P1} =0.20 V and ϕ_{P2} =0.30 V are observed on the positive branch of the cyclic voltammetry curve. These peaks may be related to the formation of Pb₃O₄ and PbO₂ on the surface of the composite electrodes. The cathodic current density peak is observed at approximately ϕ_{P3} =0.01 V on the negative potential scan due to the cathodic dissolution of PbO₂ deposited during the previous positive scan [9]. It can be also seen that in Fig. 1(c) and Fig. 1(d) the oxidation peak intensities in different electrodes do not change evidently in the S1 solution. The oxygen evolution potential changes, only showing that doping solid particles cannot change reaction mechanism of α -PbO₂ deposition [10].

3.1.2 Cyclic voltammetry curves of composite electrodes in acid solution

The cyclic voltammetry curves in 2 mol/L KNO₃ solution (pH=1.5) and in S2 solution at 60 °C are shown in Fig. 2. Figure 2(a) shows that an oxidation peak appears at 1.45 V vs SCE. This may be attributed to the solid-state surface redox transition of Pb/PbO₂. Another possible result is that the Pb²⁺ oxidizes to PbO₂ (Pb²⁺+2H₂O−2e=PbO₂+4H⁺) because it is found that the current density is rather negative at a low potential. The exponential growth of the anodic current density at potential over 1.9 V is due to oxygen evolution. The cathodic current density peak is observed at approximately 1.0 V on the negative potential scan due to the cathodic dissolution of PbO₂ deposited during the previous positive scan [11]. As shown in Fig. 2(b), the number of oxidation peaks with different electrodes does not change when the potential of oxygen evolution changes. It is shown that the doping solid particles cannot change the depositing mechanism of α -PbO₂

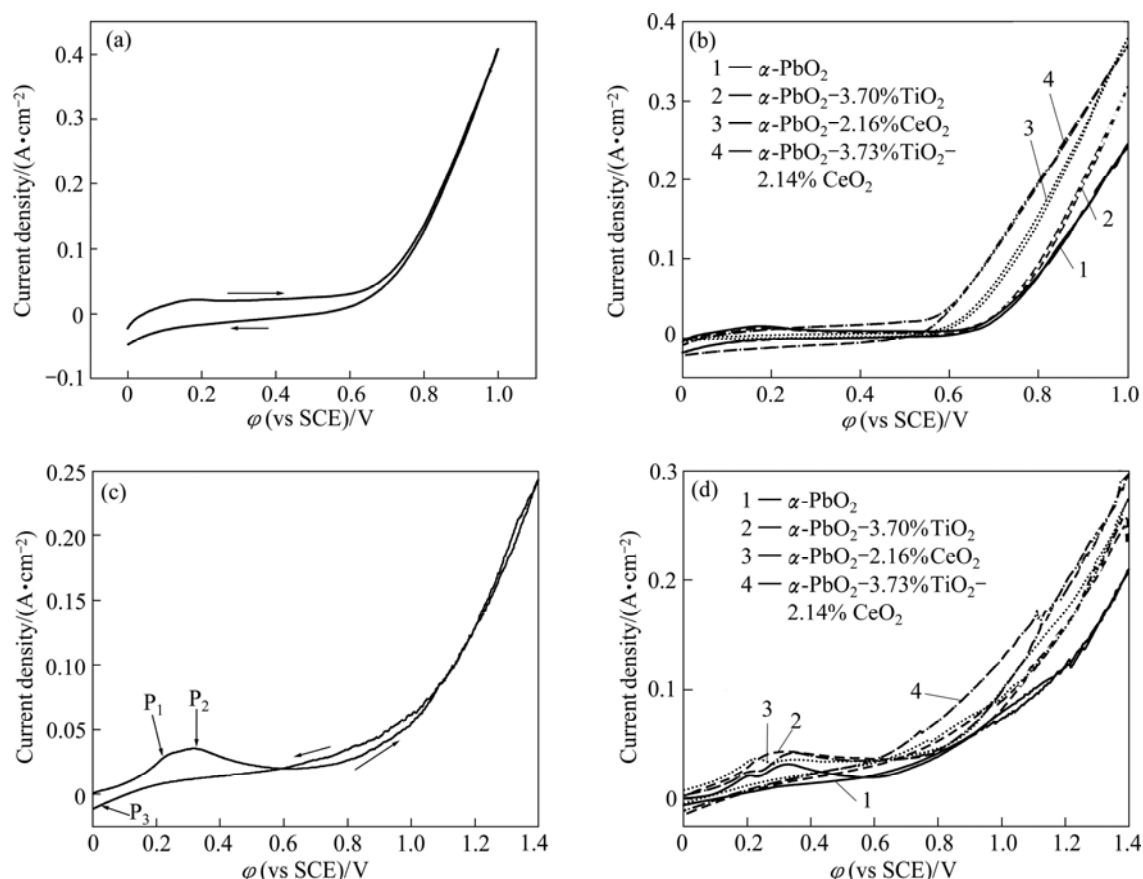


Fig. 1 Cyclic voltammetry curves in 4 mol/L NaOH solution (a, b) and in S1 solution (c, d) with scan rate of 5 mV/s

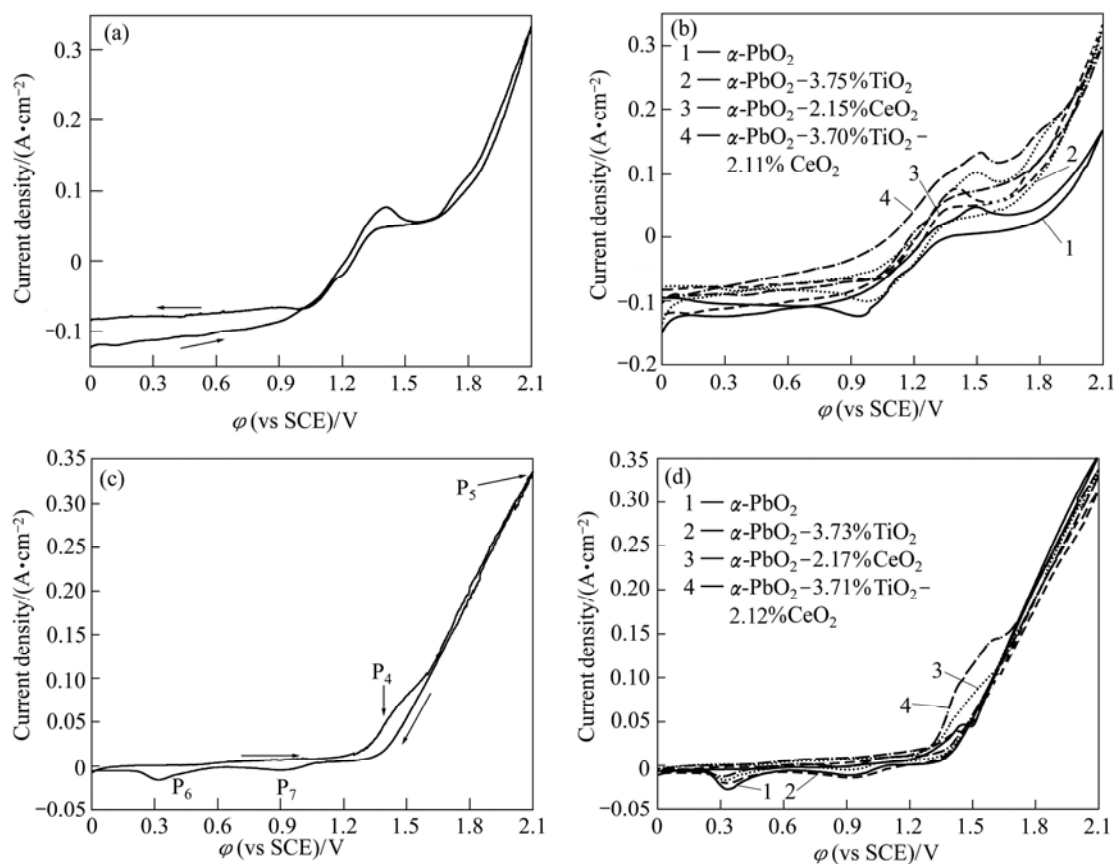
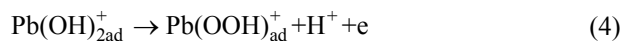
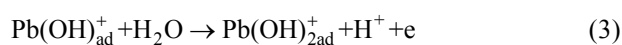


Fig. 2 Cyclic voltammetry curves in 2 mol/L KNO₃ solution (pH=1.5) (a, b) and in S2 solution (c, d) with scan rate of 5 mV/s

electrode, while the anodic current density peak at potential over 1.9 V of α -PbO₂-3.71%TiO₂-2.12%CeO₂ composite coating is higher than that of α -PbO₂. Figure 2(c) shows that the potential of the beginning deposition of β -PbO₂ is higher than 1.30 V vs SCE. Two anodic current density peaks at ϕ_{p4} =1.4 V and ϕ_{p5} =2.1 V are observed on the positive branch of the cyclic voltammetry curve. These peaks probably correspond to the formation of surface PbO₂ and O₂, respectively. The cathodic current density peaks at ϕ_{p6} =0.30 V and ϕ_{p7} =0.91 V are observed on the negative branch, and these cathodic peaks are probably due to the formation of the soluble Pb(II) and Pb(III) species. The cathodic peak at ϕ_{p3} =0.33 V does not appear in Ref. [12]. It is possible that the lower bath temperature restrains the formation of the soluble Pb(II) species. It can be also seen from Fig. 2(c) and Fig. 2(d) that the intensities of oxidize peaks of different electrodes in S2 solution do not change evidently, and only the oxygen evolution potential changes, indicating that the doped solid particles cannot change the electrodeposition mechanism of α -PbO₂. However, the cathodic current density peaks and the potential of the formation of the soluble Pb(III) species respectively decrease toward more negative with the addition of doped particles, showing that the quasi-reversible extent of electrode reaction process is large, especially the α -PbO₂-3.71%TiO₂-2.12%CeO₂ composite electrode.

The electrocatalytic activity of the electrode is determined mainly by the phase and chemical composition of the active mass. Therefore, the electrochemical deposition of lead dioxide is of great interest to achieve a desired physical and chemical property. The elucidation of the PbO₂ electrodeposition mechanism is hence very important. These data are interpreted with the following mechanism [13] (originally proposed by BECK [14]). The first step is an equilibrium hydration in aqueous solution (Eq. (1)) followed by adsorption (Eq. (2)). It follows the formation of Pb(OH)_{2ad}⁺ and Pb(OOH)_{ad}⁺ intermediate on the substrate. The final reaction ends with the production of PbO₂. These data are interpreted on the electrode surface according to the following schemes:



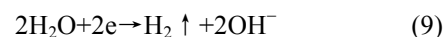
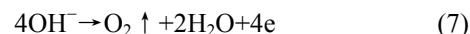
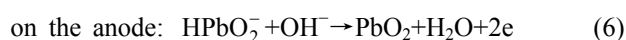
It can be seen from the CV curves in alkaline and acid solutions that two anodic peaks (except oxygen

evolution peak) are observed in the alkaline plating solution, and only one anodic peak (except oxygen evolution peak) is seen in the acid plating solution, which shows that in the acid plating solution Pb²⁺ or PbO may oxide directly to PbO₂ while in the alkaline plating solution Pb²⁺ or PbO may oxide to Pb₃O₄ first, then gradually oxide to PbO₂. This implies that in the alkaline plating solution, the first anodic peak corresponds to the formation of Pb₃O₄ and the second one corresponds to PbO₂. In other words, Pb(OH)₂⁺ and Pb(OOH)_{ad}⁺ truly exist on the surface and the further study is to make sure whether they are the soluble substance or not.

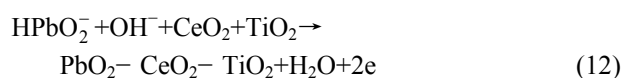
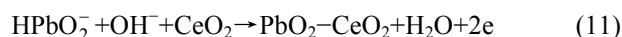
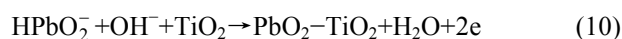
3.2 Guglielmi model of α -PbO₂ composite codeposition

The α -PbO₂ composite coatings were obtained in 4 mol/L NaOH solution saturated with litharge PbO(s) by adding nano-TiO₂ and nano-CeO₂ particles.

The main chemical reactions of electro-deposition are as follows:



TiO₂ and CeO₂ co-deposition can be described as



The co-deposition mechanism can be described by Guglielmi model shown in Fig. 3. Bilumbite ion

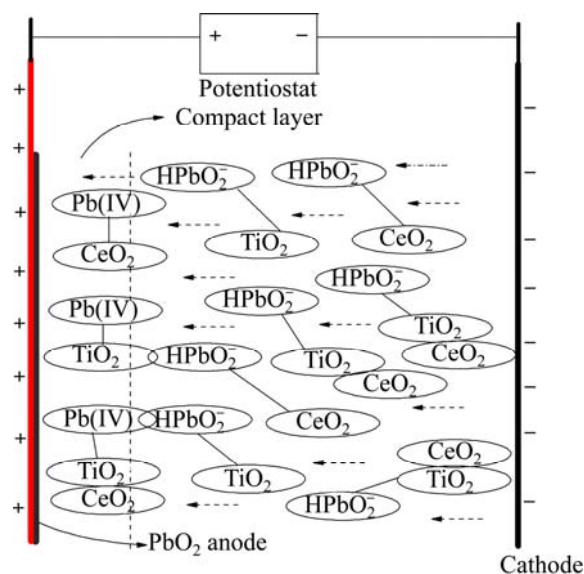


Fig. 3 Schematic diagram of Guglielmi model for TiO₂ and CeO₂ codeposition with α -PbO₂

HPbO_2^- and other ions including solvent are adsorbed on TiO_2 and CeO_2 particles, which are loosely adsorbed on $\alpha\text{-PbO}_2$ anode. Under the influence of an electric field, TiO_2 and CeO_2 particles enter the $\alpha\text{-PbO}_2$ compact layer and the oxidation of HPbO_2^- leads to a strong irreversible adsorption, which consequently makes TiO_2 and CeO_2 particles deposit along with $\alpha\text{-PbO}_2$ crystals.

3.3 Analysis of organization structure on $\alpha\text{-PbO}_2$ composite coating

Figure 4 shows the SEM images of $\alpha\text{-PbO}_2$ and $\alpha\text{-PbO}_2$ composite coating prepared on Al/conductive coating electrodes in 4 mol/L NaOH solution with addition of yellow PbO until saturation with different solid particles. Figures 4(a), (b), (c), and (d) respectively

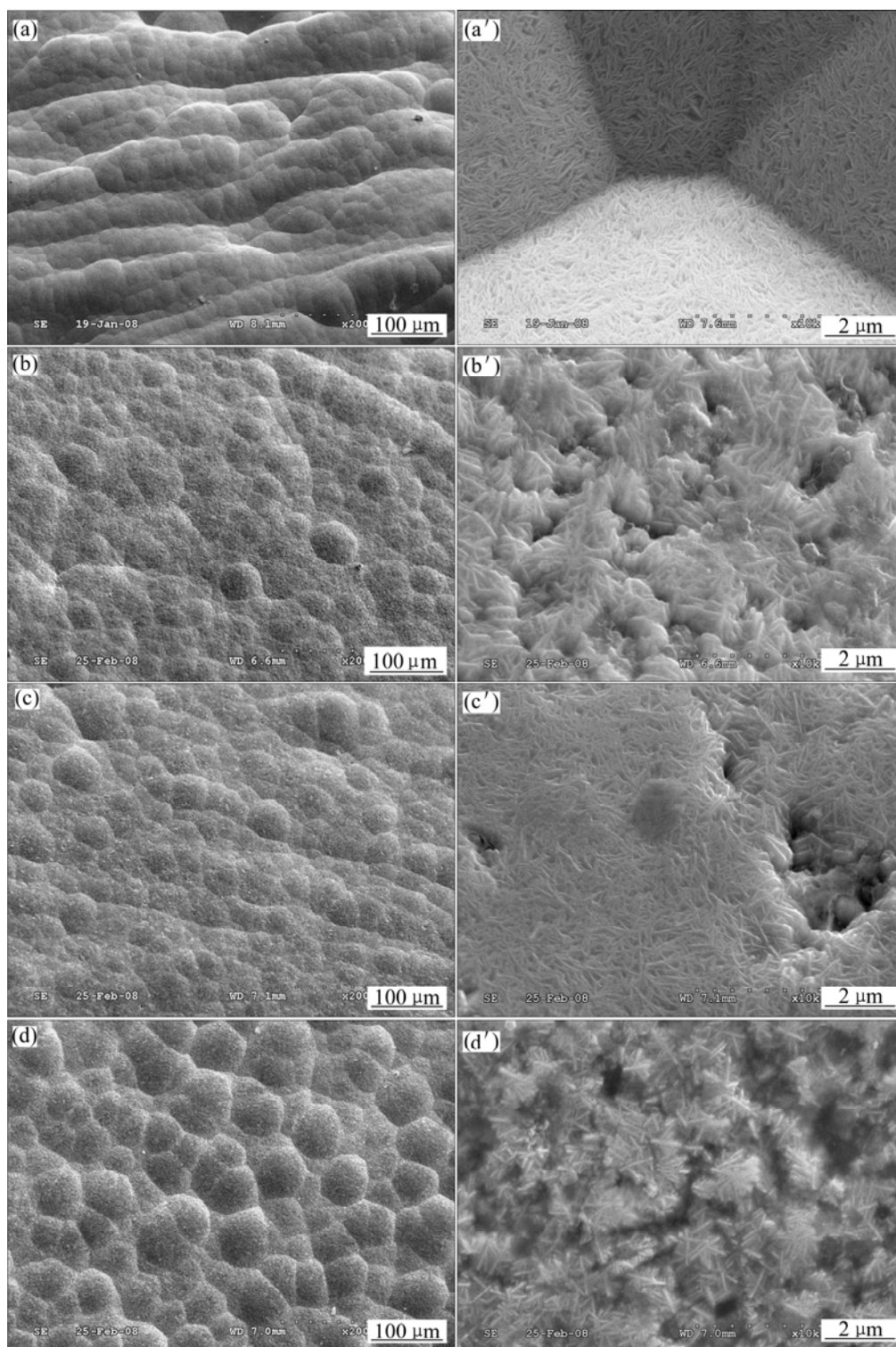


Fig. 4 SEM images of PbO_2 coatings: (a, a') pure $\alpha\text{-PbO}_2$; (b, b') Doped with TiO_2 ; (c, c') Doped with CeO_2 ; (d, d') Doped with $\text{TiO}_2\text{-CeO}_2$

correspond to the morphologies of pure α -PbO₂ coating, doped with TiO₂ (15 g/L), doped with CeO₂ (10 g/L) and doped with TiO₂(15 g/L)–CeO₂(10 g/L) composite coatings. And Figs. 4(a'), (b'), (c') and (d') show respectively the higher magnification images of Figs. 4(a), (b), (c), and (d). As shown in Fig. 4(a), there are cracks in the pure α -PbO₂ coating surface, and those cylindrical unit cells are stuck out of the surface with uneven shapes. There are no cracks in the α -PbO₂ doped with solid particles and even the surface is closely linked, especially α -PbO₂–CeO₂ composite coating has the closest and the most compact structure. The one with higher magnification reveals that the deposits are much rougher than the pure α -PbO₂, which indicates that the addition of solid particles can inhibit the growth of α -PbO₂ unit cells, and improve the specific surface area of coating surface. The increased effective area is a favourable feature in the view of a possible application as electrode materials [15]. The energy spectra of the PbO₂ coatings (from Fig. 4) are shown in Fig. 5.

Composition of coating was tested by EDAX and the different α -PbO₂ coating components are shown in Table 1. From the table we can calculate that TiO₂ mass content of TiO₂– α -PbO₂ is 3.68 %, CeO₂ mass content of CeO₂– α -PbO₂ composite coating is 2.15 % and TiO₂ and CeO₂ mass contents of α -PbO₂–TiO₂–CeO₂ composite coating are respectively 3.73 % and 2.17 %.

Table 1 Compositional analysis of PbO₂ coatings

Coating	w(Pb)/%	w(O)/%	w(Ti)/%	w(Ce)/%
Pure α -PbO ₂	88.84	11.16		
Doped with TiO ₂	83.42	14.37	2.21	
Doped with CeO ₂	84.75	13.5		1.75
Doped with TiO ₂ –CeO ₂	81.5	13.75	2.98	1.77

To avoid the thickness of PbO₂ coating affecting the phase composition, the coating thickness is controlled to be about 100 μ m by XRD using Co target and K α radiation. The phase compositions of α -PbO₂ coating doped with different particles are shown in Fig. 6 and the corresponding data are obtained with the JCPDS card. From Fig. 6, we can see that pure α -PbO₂ coating and α -PbO₂ composite coating doped with particles contain not only α -PbO₂ but also a little PbO impurity. The impurity may be obtained in the electro-deposition due to the co-deposition between insoluble Pb(OH)₂ or PbO and α -PbO₂. It is identical with the reported ones [8,16] that the strength of crystal face (200) in α -PbO₂, and the incorporation of either TiO₂ or CeO₂ particles do not affect the orienting growth of PbO₂. It can also be seen from Fig. 6 that the diffraction peak intensity reduces by the addition of particles [10]. Especially, the lowest intensity of diffraction peak of coating is obtained by the

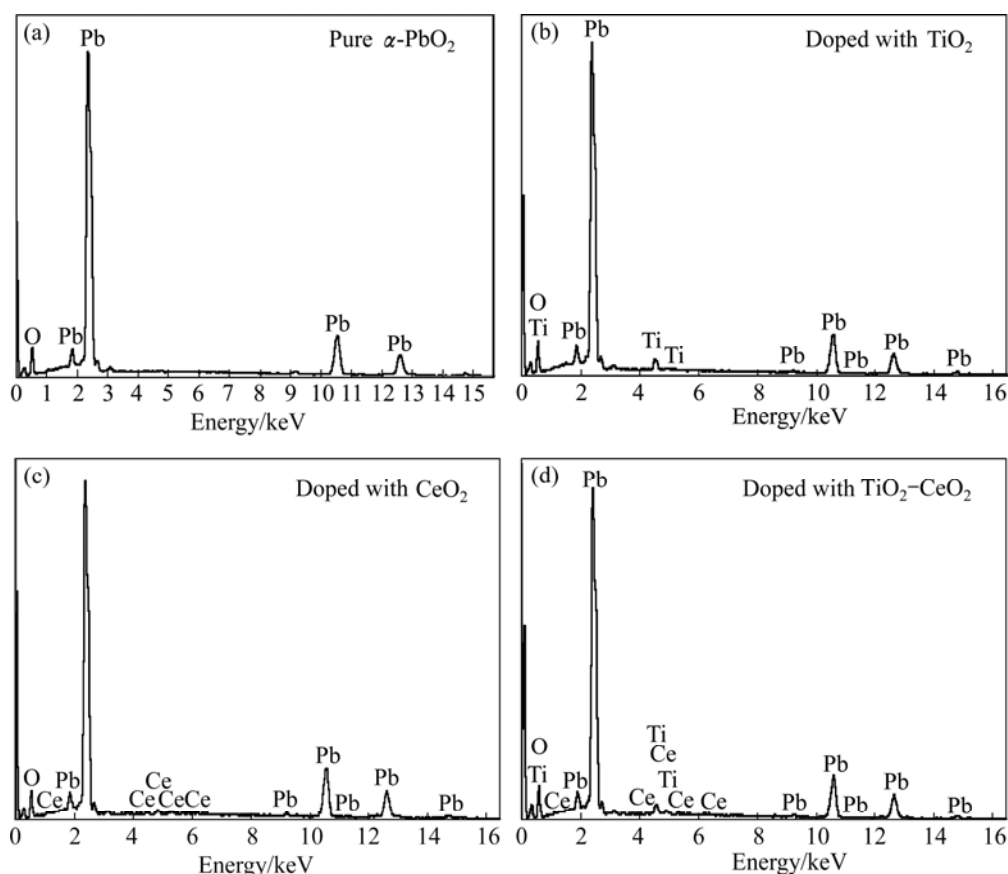


Fig. 5 EDAX spectra for different coatings: (a) Pure α -PbO₂; (b) Doped with TiO₂; (c) Doped with CeO₂; (d) Doped with TiO₂–CeO₂

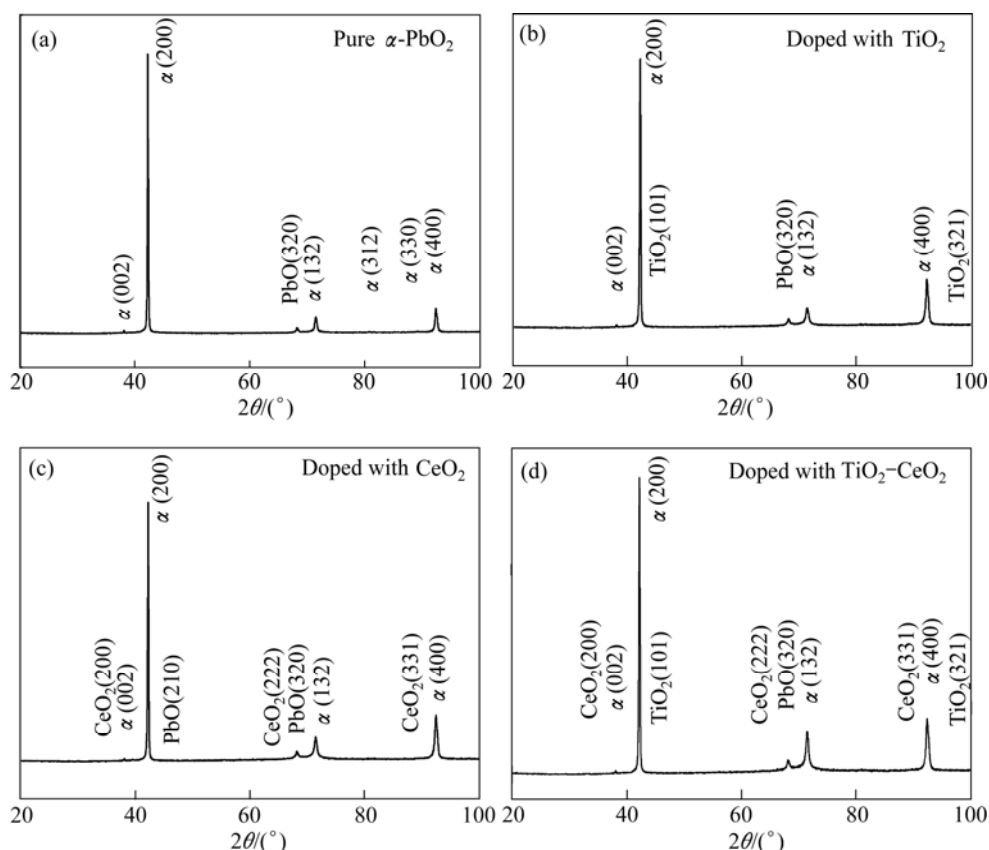


Fig. 6 X-ray diffraction patterns of different coatings: (a) Pure α -PbO₂; (b) Doped with TiO₂; (c) Doped with CeO₂; (d) Doped with TiO₂-CeO₂

addition of TiO₂ and CeO₂ solid particles together, which indicates that the addition of solid particles decreases the crystallinity of composite coating. This phenomenon can also be seen in the surface morphology shown in Fig. 4. As shown in Fig. 6, two low diffractive peaks at 42.1° and 99.7° and three low diffractive peaks at 38.6°, 69.8° and 92.2° are observed, indicating the formation of TiO₂(PDF#034-0180) and CeO₂(PDF#043-1002) on Al/conductive coating during the electrodeposition. But the contents of TiO₂ and CeO₂ are small comparable with PbO₂ film.

3.4 Anodic polarization curves

Figure 7 shows the anodic polarization curves of Al/conductive coating/ α -PbO₂, Al/conductive coating/ α -PbO₂-3.73%TiO₂, Al/conductive coating/ α -PbO₂-2.17%CeO₂ and Al/conductive coating/ α -PbO₂-3.71%TiO₂-2.12%CeO₂ electrodes in 50 g/L Zn²⁺, 150 g/L H₂SO₄ solution at 25 °C. The anodic polarization curve of Pb electrode is also shown for comparison. From Fig. 7, it is apparent that the electrocatalytic activity of the Al/conductive coating/ α -PbO₂ is much superior to that of Pb for the anodic evolution of oxygen. It can also be seen that the potential of Al/conductive coating/ α -PbO₂-3.71%TiO₂-2.12%CeO₂ anode is the lowest at a constant current density, which shows that the

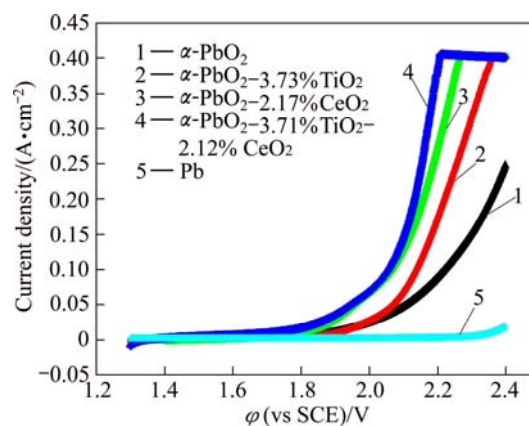


Fig. 7 Polarization curves of different electrodes in Zn²⁺ 50 g/L, 150 g/L H₂SO₄ at 25 °C

electrocatalytic activity of the anode is the best. The potential of Al/conductive coating/ α -PbO₂-2.17%CeO₂ anode is lower than that of Al/conductive coating/ α -PbO₂-3.73%TiO₂, which indicates that the electrocatalytic activity of electrode doped CeO₂ is better than that of electrode doped with TiO₂. The potential of Al/conductive coating/ α -PbO₂ anode is the highest, which indicates that the electrocatalytic activity of electrode is improved evidently with addition of particles.

4 Conclusions

1) CV curves measurement shows that doping solid particles cannot change the reaction mechanism of coating in acid or alkaline plating solution but they can improve the deposition rate and reduce the reaction potential. Based on the mechanism of electro-deposition, Pb_3O_4 oxidative peak appears in alkaline bath but in acid.

2) The physicochemical properties of the composites significantly differ from those of lead dioxide and are determined by the compositions of these materials. SEM and XRD analyses show that doping solid particles can inhibit the growth of α - PbO_2 unit cell, and improve specific surface area of coating surface. The surface roughness of α - PbO_2 - TiO_2 - CeO_2 composite coating is the highest, and the diffraction peak intensity of the coating with doped solid particles is lower than that of pure α - PbO_2 coating. Especially, the lowest of the diffraction peak intensity of the coating is obtained with addition of TiO_2 and CeO_2 .

3) The α - PbO_2 -3.72% TiO_2 -2.12% CeO_2 electrode exhibits the best electrocatalytic activity for O_2 evolution, and the Guglielmi model for CeO_2 and TiO_2 codeposition with α - PbO_2 is also proposed.

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α - PbO_2 - CeO_2 - TiO_2 复合电极材料的电合成及物化性能

陈步明¹, 郭忠诚^{1,2}, 徐瑞东¹

1. 昆明理工大学 冶金与能源工程学院, 昆明 650093; 2. 昆明理工恒达科技有限公司, 昆明 650105

摘要: 采用电化学阳极复合电沉积技术, 将纳米 TiO_2 和 CeO_2 颗粒加入氧化铅中并溶入 4 mol/L NaOH 溶液中至饱和, 在铝基体上制备 α - PbO_2 - CeO_2 - TiO_2 复合镀层。利用循环伏安(CV)、扫描电镜(SEM)、能谱(EDAX)和 X 射线衍射(XRD)分析复合电极的沉积机理、表面形貌、成分及结构。结果表明: 在酸、碱性镀液中, 掺杂固体颗粒不会改变 α - PbO_2 电极的反应机理, 但能提高沉积速率和降低析氧电势。掺杂固体颗粒能抑制 α - PbO_2 晶胞的长大, 增大镀层的比表面积, 且掺杂固体颗粒的复合镀层的衍射峰强度比未掺杂的 α - PbO_2 镀层的低很多。 α - PbO_2 -2.12% CeO_2 -3.71% TiO_2 复合电极材料的析氧催化活性最好。提出了两种颗粒的复合共沉积模型。

关键词: 铝; 二氧化铅; 复合电极; 电催化; 物化性能

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