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The influence of impurities on Ga electrowinning: Vanadium and iron

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ARTICLE INFO

Article history: Received 26 August 2013 Received in revised form 29 January 2014 Accepted 8 March 2014 Available online 27 March 2014

Keywords: Gallium Vanadium Iron Electrowinning Current efficiency

1. Introduction

The compound semiconductors of gallium are widely used in optoelectronic devices such as advanced semiconductor, LEDs and DVDs (Moskalyk, 2003). Recently, many researchers also pay great attention to gallium-containing film materials, such as Cu(InGa)Se₂ (CIGS) thin film (Bhattacharya and Fernandez, 2003; Bhattacharya et al., 2000; Zhang et al., 2003), Fe–Ga alloy (Iselt et al., 2011; McGary et al., 2010) and GZO film (Yamamoto et al., 2012; You and Hua, 2012). It is suggested that gallium is promising in photovoltaic industry in the future. Therefore, the demand of gallium will steadily increase.

Gallium is extracted primarily from mother solution of alumina production. Many methods have been developed to extract gallium from Baver liquor (Zhao et al., 2012). Ion exchange by resin is considered as the best method to enrich Ga from Bayer liquor because of high recovery rate, easy operation, and non-effect on alumina process (Riveros, 1990). Then, electrowinning is carried out to obtain metal Ga from solution with Ga concentration of 2-3 g/L. At present, most of Ga is produced by the combination technology of ion exchange and electrowinning all over the world. However, metal impurities such as Fe and V usually exist in aluminate liquids. The coextraction of impurities and Ga is a major issue for the combination technology (Bautista, 2003). V is absorbed by resin and then transferred into Ga solution for electrowinning. In addition, Fe from Bayer liquor, raw material and equipment corrosion is also accumulated in Ga solution. It is found that Fe or V inhibited gallium electrodeposition during direct electrochemical extraction of gallium on liquid mercury cathode (Radvila, 1983). The

ABSTRACT

During the electrolytic extraction of Ga, iron and vanadium are main metal impurities in Ga solution. The influences of Fe(III) and V(V) on current efficiency (*QE*) of Ga electrowinning were studied. The surface structures of electrodeposited Ga coatings were examined by SEM, EDS and potentiodynamic polarization. The results indicate that *QE* is sharply decreased in the presence of V(V). Spherical Ga particles are easily produced in Ga solution with impurities, especially in the presence of V(V) and at higher current density. It is difficult for Ga particles with impurities and newly formed Ga crystals to incorporate into Ga films due to the difference of surface structure and properties between Ga and impurities. As a result, newly formed Ga crystals are easily re-dissolved which leads to the decrease of *QE*. When both Fe(III) and V(V) exist in Ga solution, *QE* at lower current density is increased due to the siderophile affinity of Ga.

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reason was ascribed to the formation of thin oxide films on cathode surface. However, other researchers thought that high catalytic activity of V for hydrogen evolution reaction (HER) was unfavorable to gallium electrowinning (Dorin and Frazer, 1988). Both V and Fe are deleterious for gallium electrodeposition. However, the relatively comprehensive effects of V and Fe on gallium deposition and the influence mechanism are not clear. It is necessary to study the influence regulation and mechanism of V and Fe on gallium electrowinning in details.

It is showed that V(V) is the predominant oxidation state in alkaline solutions (Riveros, 1990). In present work, the effects of V(V) and Fe(III) on current efficiency (*QE*) of Ga electrodeposition from alkaline solution were studied systematically. In order to understand the influence mechanism of the impurities, surface morphology, chemical composition and electrochemical property of gallium deposits were examined.

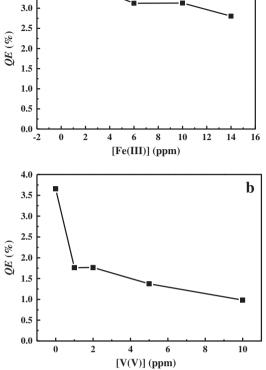
2. Experimental details

Both cathode and anode were 316L stainless steel for gallium electrowinning. The geometrical area of electrode was 30 cm², and electrode spacing was 4 cm. Electrodes were firstly polished using 800 and 1200 emery paper, and then immersed in 5% sulfuric acid for 10 min and cleaned in ultrasonic subsequently. In order to avoid the influence of steel substrates and investigate gallium deposition process in the presence of impurities, Ga-coating cathodes were adopted and prepared as follows: Ga coatings were pre-electrodeposited on stainless steel electrodes in the solution with 2 g/L gallium and 10 wt.% NaOH at 0.1 A·cm⁻² for 1 h.

Solution for Ga electrowinning consisted of 10 wt.% NaOH and 1–2 g·L⁻¹ Ga(III). An Fe(III) solution of 1.5 g·L⁻¹ was prepared by dissolving Fe₂(SO₄)₃ in water. A V(V) solution of 1.5 g·L⁻¹ was prepared

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4.0

3.5

Fig. 1. The effects of Fe (a) and V (b) on *QE* in 10% NaOH and 1 g·L⁻¹ Ga(III) solution. Temp, 40 °C; deposition time, 3 h; current density, 0.1 A·cm⁻².

by dissolving NaVO₃ in water. A certain volume of Fe(III) or V(V) stock solutions was added into Ga solution to adjust impurity concentration. The concentrations of Ga, Fe and V in solution were analyzed by an Inductivity Coupled Plasma-Optical Emission spectrometer (ICP-OES, PE Optima 5300DV). Fe(III) mainly exists in alkaline media in the form of Fe(OH)⁻₄ with low solubility (Allanore et al., 2007). The reaction equilibrium is

$$Fe_2O_3 + 3H_2O + 2OH^- = 2Fe(OH)_4^-.$$
(1)

The electrochemical reduction or adsorption of iron hydroxyl anion on electrode leads to the dissolution of ferric oxide into alkaline solution according to Eq. (1).

Current efficiency (*QE*) of Ga electrodeposition was analyzed as follows:

$$QE = \frac{\text{Actual weight of gallium by electroreduction}}{\text{Theoretical weight of gallium by Faraday's Law}} = \frac{(C_0 - C_e) \times V}{(It/nF) \times M}$$
(2)

where C_0 and C_e are Ga concentrations before and after Ga electrodeposition, respectively; *V* is solution volume; *I* is current intensity; *t* is electrolytic time; *n* is number of electrons participating in the reaction; *F* is Faraday constant, 96,485 C·mol⁻¹; and *M* is molar mass of gallium, 69.7 g·mol⁻¹. Ga concentrations before and after electrolysis are analyzed by ICP-OES.

Potentiodynamic polarization curves were measured on CHI 604B electrochemical working station (CH Instrument, Inc.) with conventional three-electrode cell in 10% NaOH solution. Pt foil and saturated calomel electrode (SCE) were used as counter electrode and reference electrode, respectively. The working electrodes were Ga-coating

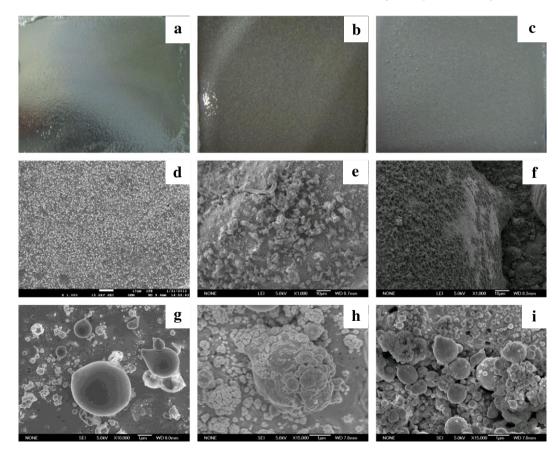


Fig. 2. Photos and SEM images of electrodeposited gallium. (a, d, g) in the solution without impurity; (b, e, h) in the solution with 4 ppm Fe(III); (c, f, i) in the solution with 2 ppm V(V). Ga(III) concentration, 1 g·L⁻¹; temp, 40 °C; deposition time, 3 h; current density, 0.1 A·cm⁻².

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